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INSTITUTE OF MATERIALS SCIENCE AND ENGINEERING
ÚSTAV MATERIÁLOVÝCH VĚD A INŽENÝRSTVÍ

ADVANCED METALLOGRAPHIC TECHNIQUES FOR ALUMINUM AND ITS ALLOYS
POKROČILÉ METODY PŘÍPRAVY VZORKŮ AL A JEHO SLITIN

BACHELOR'S THESIS
BAKALÁŘSKÁ PRÁCE

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VEDOUCÍ PRÁCE

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BRNO UNIVERSITY OF TECHNOLOGY
FACULTY OF MECHANICAL ENGINEERING

Assignment Bachelor's Thesis

Institut:	Institute of Materials Science and Engineering
Student:	**David Rychlý**
Degree programm:	Applied Sciences in Engineering
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Supervisor:	**Ing. Mgr. Šárka Mikmeková, Ph.D.**
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As provided for by the Act No. 111/98 Coll. on higher education institutions and the BUT Study and Examination Regulations, the director of the Institute hereby assigns the following topic of Bachelor's Thesis:

Advanced metallographic techniques for aluminum and its alloys

Brief Description:

The thesis focuses on the metallography and microstructures of pure aluminum and aluminum alloys. Aluminum is a soft metal with a face–centered cubic crystal structure. Its softness makes it difficult to prepare and a sensitivity to mechanical deformation generates mechanical twins or Neumann bands. Aluminum is very corrosion resistant and a thin oxide film form on a freshly polished surface. This film is responsible for good corrosion resistence, but also makes etching difficult. The thesis goal is to test various etchants and etching conditions in order to establish optimal conditions for selected types of Al alloys. The surface quality will be analyzed by a optical, a laser, and a electron microscopy. Al alloys will be provided by prof. Kenji Matsuda (Toyama University, Japan).

Bachelor's Thesis goals:

- metallography of Al and its alloys – an overview and recent trends
- test of standard etchants and optimizing of etching conditions
- surface quality analysis by light, laser, and electron microscope
- establishing of optimal sample preparation technique for selected Al alloys

Recommended bibliography:

GRAFF, W. R. and D. C. SARGENT. A new grain-boundary etchant for aluminum alloys [short communication]. Metallography 14, 69–72, 1981.

Metallographic Techniques for Aluminum and Its Alloys. VANDER VOORT, George F., ed. Metallography and Microstructures [online]. ASM International, 2004, 2004, s. 711-751 [cit. 2020-1--03]. ISBN 978-1-62708-177-1. Dostupné z: doi:10.31399/asm.hb.v09.a0003769

YANG, H. S. A new light optical metallographic technique for revealing grain structures of common 2000, 5000, and 7000 series aluminum alloys. Mater. Charact. 38, 165–175, 1997.

Deadline for submission Bachelor's Thesis is given by the Schedule of the Academic year 2020/21

In Brno,

L. S.

prof. Ing. Ivo Dlouhý, CSc. Director of the Institute	doc. Ing. Jaroslav Katolický, Ph.D. FME dean

Abstrakt

Hlavním cílem této práce je optimalizovat metalografickou přípravu hliníku pro specifické potřeby rastrovací elektronové mikroskopie. Rastrovací elektronová mikroskopie je velice citlivá na kvalitu připraveného povrchu a základní metody přípravy užívané pro světelnou mikroskopii jsou nedostatečné. Práce se zaměřuje na optimalizaci a hledání nových metalografických metod, které splňují vysoké požadavky rastrovací elektronové mikroskopie. Povrch musí být připraven co nejkvalitněji, bez defektů a artefaktů z přípravy. Jako testovací materiál pro experimenty byl vybrán čistý hliník. Základní a pokročilé metody přípravy byly experimentálně testovány a nejvíce slibné postupy poté optimalizovány. Několik různých variant přípravy povrchu bylo vyzkoušeno v rámci metod koloidních silik a elektrolytického leštění. Připravený povrch byl následně zkoumán pomocí světelné mikroskopie, rastrovací elektronové mikroskopie s nízkými urychlovacími napětími a konfokální mikroskopie. Závěrem byl stanoven optimální metalografický postup vyhovující vysokým požadavkům elektronové mikroskopie a zajišťující připravený povrch s minimem defektů.

Abstract

Main goal of this thesis is to optimize the metallographic preparation process of aluminium for specific needs of a scanning electron microscopy (SEM). The SEM is highly sensitive to surface quality and conventional sample preparation methods used for an optical microscopy (OM) become insufficient. The thesis focuses on optimizing and finding novel metallographic methods fulfilling high requirements of the SEM imaging. It requires perfect quality surface without any damage, such as preparation artifacts and scratches. A pure aluminium has been selected as an experimental material. Conventional and advanced metallographic techniques were tested and the most promising techniques were optimized. Several surface finish techniques were examined in order to obtain a perfect quality surface, such as colloidal sillica polishing and electropolishing. The surface quality was examined by the OM, the low voltage SEM, and a confocal microscopy. Finally, the metallographic technique providing the aluminium surface with minimum defects is established.

Klíčová slova

Hliník, Metalografie, Rastrovací elektronová mikroskopie, Mechanicko-Chemické leštění, Elektrolytické leštění

Keywords

Aluminium, Metallography, Scanning electron microscopy, Mechanical-Chemical polishing, Electrolytic polishing (Electropolishing)

Rozšířený abstrakt

Práce je zaměřena na metalografickou přípravu vzorků hliníku pro rastrovací elektronovou mikroskopii. Rastrovací elektronová mikroskopie klade veliký důraz na kvalitu pozorovaného povrchu a základní techniky, dostačující pro zkoumání povrchu světelnou mikroskopií, jsou nedostatečné. Náplní práce je tedy hledání a optimalizace základních metod přípravy hliníku, na které následně navazuje optimalizace pokročilých metod jako jsou koloidní siliky a elektrolytické leštění.

Hliník je po oceli druhým nejpoužívanějším strojírenským materiálem. V přírodě se vyskytuje ve formě bauxitu a za pomocí Bayerova a Halt-Heroultova procesu je z této rudy získáván. Ve své čisté podobě má hliník teplotu tání 660 °C a pod touto teplotou tuhne do FCC krystalické mřížky, díky níž má nízké pevnostní charakteristiky. Toto má za následek jeho obtížnější metalografickou přípravu. Má však vynikající korozivzdornost, recyklovatelnost, nízkou hustotu (2700 kg/m^3), velmi dobrou elektrickou a tepelnou vodivost a také není toxický a zdraví škodlivý. Čistý hliník je převážně využíván na dráty vysokého napětí a dříve v elektrotechnice. Přidáním legur jako měď, zinek, hořčík, křemík, mangan nebo lithium vzniká rozmanitá škála slitin, které mění zejména jeho mechanické, korozní a elektrické vlastnosti. Slitiny Al-Cu, Al-Mg-Si a Al-Zn jsou vhodné pro tepelné zpracování, konkrétně precipitační vytvrzování. Tepelně zpracovávané slitiny nachází své využití zejména v leteckém a kosmickém průmyslu. Slitiny Al-Mn, Al-Si a Al-Mg nejsou vhodné pro tepelné zpracování a je třeba je tvářet za studena, pokud chceme zlepšit jejich mechanické vlastnosti. Slitiny tvářené za studena mají využití jako výměníky tepla, kuchyňské potřeby, svařovací dráty, v elektrotechnice nebo při stavbě budov. [1][3][4]

Metalografie je oblast vědy zabývající se přípravou povrchu pro následné pozorování za účelem zjišťování informací. Na základě kvalitně připraveného povrchu získáváme za pomocí mikroskopie kvalitativní a kvantitativní informace o materiálu. Mezi kvalitativní analýzu patří např. podoba fází a prvků vyskytujících se v materiálu. Kvantitativní analýza nám dává informace o velikosti zrn, poměru, v jakém jsou fáze ve slitině vůči sobě apod. Základem metalografie je mikrostruktura. Výsledek, kterého chceme dosáhnout je mikrostruktura bez artefaktů jako např. škrábance, deformace, kontaminace, mikrotrhliny apod. [5][6]

Metalografická příprava je komplexní proces, který záleží na mnoha krocích a parametrech a je třeba vysoká opatrnost, aby vzorky nebyly kontaminovány. Prvním krokem je volba místa odběru vzorku a jeho následné uříznutí. Po odebrání je vzorek zalit do pryskyřice pro lepší manipulaci. Následně je vzorek nejprve mechanicky broušen, většinou kombinací brusných kotoučů s vodou. Po brusných kotoučích následují kotouče látkové s využitím diamantových past se zrny malé velikosti. Tuto kombinaci brusných kotoučů a látkových kotoučů s diamantovými pastami hromadně nazýváme mechanickým leštěním. Po základním mechanickém leštění je možno přistoupit k pokročilým metodám přípravy vzorků. První z nich je mechanicko-chemické leštění koloidními silikami. Koloidní siliky jsou kapalné suspenze velmi jemných částic, které povrch mechanicky leští a zároveň s ním chemicky reagují. Až 50 % těchto suspenzí je tvořeno pevnými částicemi, avšak stále zachovávající vlastnosti kapaliny. Další pokročilou metodou pro přípravu povrchu je elektrolytické leštění. Tato metoda spočívá ve vytvoření elektrolýzy mezi leštěným vzorkem jako anodou a nástrojovou elektrodou jako katodou ve zvoleném elektrolytu. Jedná se tedy o kontrolované rozpouštění povrchu vzorku, které nezanechává žádné mechanické deformace. Jedná se o velice experimentální metodu závislou na spoustě parametrů jako je volba a teplota elektrolytu, napětí a čas. [2][5][8][9][10]

Po přípravě povrchu následuje zkoumání povrchu. Pro tyto účely poslouží světelná, konfokální a elektronová mikroskopie. Světelná mikroskopie je metoda pozorování založená na zvětšení detailu za pomocí viditelného světla. Tohoto zvětšení je dosaženo soustavou čoček v objektivu a okuláru, které je následně pozorováno buď přímo lidským okem nebo promítáno na plochu počítače. Konfokální neboli laserová mikroskopie spočívá ve filtrování informací, které nejsou v rovině pozorování a negativně ovlivňují optický obraz. Konfokální mikroskopy také umožňují měření drsnosti povrchu tím, že sejmou data z několika rovin a na základě nich zprostředkují 3D profil povrchu, ze kterého je možné určit jeho drsnost. Pro větší zvětšení, kde už nestačí světelná mikroskopie využijeme rastrovací elektronovou mikroskopii. Elektronová mikroskopie spočívá v interakci elektronového paprsku s povrchem vzorku. Elektrony jsou uvolněny ze zdroje, urychlovány urychlovacím napětím a následně přes soustavu cívek hnány na povrch pozorované součásti, která je ve v komoře s vakuem. Na základě interakce rozlišujeme elektrony na zpětně odražené a sekundární. Z těchto informací poté vzniká obraz s vysokým zvětšením odhalující detail mikrostruktury. Elektronový mikroskop je také schopen elektronové disperzní spektroskopie (EDS). Při interakci elektronu s povrchem je vyzařováno rentgenové záření pro každý prvek specifické. EDS analýza porovnává vyemitované záření na součásti s etalony známých prvků a dává tak informaci o chemickém složení vzorku. [16][20]

Jako vzorky pro experimentální část byl zajištěn hliník s čistotou 99,5 %. Tato skutečnost byla ověřena za pomocí EDS analýzy v elektronovém mikroskopu. Zbylé 0,5 % jsou prvky ve formě precipitátů, zejména železo a křemík. Tyto precipitáty mají podobu jehlic a jsou označovány jako β-AlFeSi. Vznikají díky malé rozpustnosti železa v hliníku, které se do vzorku dostalo pravděpodobně při metalurgickém procesu. Díky svojí jehlicovité struktuře zhoršují mechanické vlastnosti. Precipitáty lze vidět na Fig. 1.

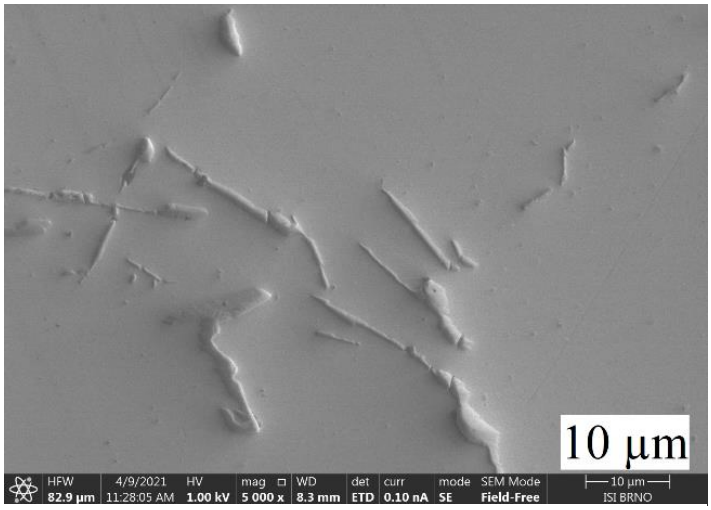


Fig. 1 - 5000x zvětšení, β-AlFeSi precipitáty

Na základě dostupné literatury a doporučených postupů byly vybrány 4 procesy mechanické přípravy pro experimentální část, na které následně navázala příprava koloidními silikami a elektrolytickým leštěním. Mechanická příprava sestávající z broušení a leštění diamantovými pastami způsobila poškrabání povrchu a je tím pádem nevyhovující pro elektronovou mikroskopii. Následný přechod na pokročilou metodu koloidních silik nám ukázal, že OP-U suspenze od firmy Struers kontaminuje a degraduje povrch vzorků, zatímco suspenze OP-S jej připraví velmi dobře. Také se ukázalo, že použití koloidních silik produkuje lepší výsledky při použití delších časů, konkrétně až 30 minut. Druhou testovanou pokročilou metodou bylo elektrolytické leštění. Experimenty nám ukázaly, že stačí vzorek připravit mechanicky a poté rovnou přejít na elektrolytické leštění. Není třeba zařazovat mezi tyto dvě metody i metodu koloidních silik. Jako optimální elektrolyt byl zvolen komerčně dostupný elektrolyt A2 od firmy Struers. Napětí 25 V a časy 15-25 sekund vyšly jako optimální parametry pro nejlepší kvalitu připraveného povrchu.

Metoda leštění pomocí koloidních silik i metoda elektrolytického leštění se ukázaly být dobrým nástrojem pro přípravu hliníku pro účely elektronové mikroskopie. Proces leštění pomocí koloidních silik je více časově náročný, ale neklade požadavek na to, aby laboratoř vlastnila elektrolytickou leštičku. Elektrolytické leštění je z hlediska času mnohem rychlejší, ale vyžaduje další přístroj do metalografické laboratoře. V tabulce 1 a 2 jsou specifikované výsledné optimalizované procesy pro přípravu čistého hliníku pro elektronovou mikroskopii.

Optimální proces koloidních silik:

Table 1 - Upravený Proces 4: Struers Metalog Guide A + koloidní siliky

Krok	**Brusný papír**	**Voda**	**Lubrikant**	**Suspenze**	**Zatížení [N]**	**Čas**	**Rychl. disku [rpm]**	**Rychl. hlavy [rpm]**	**Rotace**
1	SiC 220 Grit	Ano	-	-	20	60 s	300	150	Sousl.
2	MD-Largo	-	-	DiaDuo 9 µm	10	180 s	150	100	Sousl.
3	MD-Mol	-	IPA	DP Paste P 3 µm	5	240 s	200	100	Sousl.
4	MD-Dac	-	IPA	DP Paste P 1 µm	5	120 s	200	100	Sousl.
5	MD-Dac	-	IPA	DP Paste P 0,25 µm	5	120 s	200	100	Sousl.
6	MD-Chem	-	-	70% OP-S 30% H2O	5	30 min	150	100	Nes.

Optimální proces elektrolytického leštění:

Table 2 - Upravený Proces 4: Struers Metalog Guide A + elektrolytické leštění

Krok	**Brusný papír**	**Voda**	**Lubrikant**	**Suspenze**	**Zatížení [N]**	**Čas**	**Rychl. disku [rpm]**	**Rychl. hlavy [rpm]**	**Rotace**
1	SiC 220 Grit	Ano	-	-	20	60 s	300	150	Sousl.
2	MD-Largo	-	-	DiaDuo 9 µm	10	180 s	150	100	Sousl.
3	MD-Mol	-	IPA	DP Paste P 3 µm	5	240 s	200	100	Sousl.
4	MD-Dac	-	IPA	DP Paste-P 1 µm	5	120 s	200	100	Sousl.
5	MD-Dac	-	IPA	DP Paste-P 0,25 µm	5	120 s	200	100	Sousl.
6	Electropolishing = A2 elektrolyt, 22 °C, 25 V, 15-25 s, Flow rate 10, Plocha 1 cm^2								

RYCHLÝ, David. *Pokročilé metody přípravy vzorků Al a jeho slitin.* Brno, 2021. Dostupné také z: https://www.vutbr.cz/studenti/zav-prace/detail/132529. Bakalářská práce. Vysoké učení technické v Brně, Fakulta strojního inženýrství, Ústav materiálových věd a inženýrství. Vedoucí práce Šárka Mikmeková.

I hereby declare that I wrote bachelor's thesis „Advanced metallographic techniques for aluminium and its alloys" all by myself under the direction of my supervisor, Ing. Mgr. Šárka Mikmeková, Ph.D. All used sources are listed in the used literature chapter.

In Brno 19.5.2021 David Rychlý

I would like to express my deepest gratitude to my supervisor Ing. Mgr. Šárka Mikmeková, Ph.D. for valuable insight and guidance throughout the process of writing this thesis. I would also like to thank Ing. Ondřej Ambrož for his helpful assistance with the experiments. Lastly, I would like to thank my mother, who set me on this path and supported me all the way.

Table of contents

1 Introduction

1.1 Al and its alloys

Aluminium is highly reactive element. In nature, it usually occurs in compounds, the primary source being Bauxite $Al_2O_3 \cdot 2\ H_2O$. Using Bayer process, Aluminium oxide (Alumina) is obtained from Bauxit. After applying the Bayer process, Alumina is brought through Electrolytic cells procedures and Hall-Heroult process with the final product being pure aluminium. [1]

One of the main economical features of aluminium is its recyclation. Firstly, recycling one tonne of Aluminium takes just 5 % of energy of the production of new tonne of aluminium. Secondly, there is no difference between newly produced and recycled aluminium in its quality, making it reusable. [1]

Aluminium can be used either pure or in alloys. Pure aluminium is good thermal and electrical conductor, corrosion resistant and ductile. Nevertheless, it‘s also very soft and has weak mechanical properties compared to steel. Another big advantage is its density, being 2700 kg/m^3, which is almost 3times less than density of steel (7850 kg/m^3). It has better strength to weight ratio than steel, making it suitable for transport industries, like aviation or automotive industries. Aluminium has low melting temperature, standing at 660°C. Below this temperature, it crystallizes in FCC lattice, therefore being non-polymorphic. This FCC lattice is the main reason, why aluminium is so soft and low strength in pure form. [1]

Above mentioned weak mechanical properties, like low strength and hardness make metallographic preparation difficult, but is fair when compared to preparation of magnesium or titanium, which are sensitive to mechanical deformations like twins or Neumann bands. Its high reactivity also gives it high corrosion resistance, because creation of thin Al_2O_3 film on surface causes difficulty during etching. It usually has high concentration of intermetallic precipitates, which may produce small improvement of properties, but also might be detrimental. [1][2]

Aluminium is also very good thermal and electrical conductor. Its thermal conductivity is 3times higher than steel, finding good use in products like heat exchangers or kitchen equipment. Aluminium owes its high use in the culinary industry to its non-toxic behavior. Electrical conductivity is also very good, making it second most used conductor material after copper. It also possesses excellent reflectivity of radiant energy. This is used for insulation purposes either in house construction or as popular material for camping sleeping pads. Aluminium alloys are suitable for heat treatment such as homogenization, annealing, precipitation/age hardening and many more. When it’s not possible to heat treat it, it is usually hardened by procedures like cold rolling. [1]

Aluminium is the second most used element after steel in the engineering industry. Its use ranges from castings to thin foils across many other industries. Aluminium, however, finds much broader use as part of alloys. Most common components in Al alloys are copper, zinc, magnesium, silicon, manganese and lithium. Also, minor components like chromium, titanium, lead, bismuth, nickel or zirconium can alternate mechanical properties. Very small amounts of iron are also inevitable in aluminium. Aluminium alloys are summarized on Table 2. [1][3][4]

Table 3 - Summary of aluminium alloys

Symbol	Alloying element	Information	Use + example
1xxx	None (99 %+ Al) -> Pure aluminium	Pros: corrosion resistance, electrical/thermal conductivity Cons: Low hardness, low strength	Transmission/power grids (1350) Food trays (1100)
2xxx	Cu	Pros: Combination of high strength and toughness Cons: Lower corrosion resistance (solved by painting or cladding by 6xxx)	Aircraft alloys (2024)
3xxx	Mn	Moderate strength, good workability	General purpose alloys, heat exchangers, cooking utensils (3000) Beverage cans (3004)
4xxx	Si	Si lowers the melting point of Al without introducing brittleness.	Welding wires (4043) Brazing alloys
5xxx	Mg	Moderate to high strengths, good weldability and corrosion resistance, water corrosion resistance	Electronics (5052) Marine applications (5083) Building constructions
6xxx	Mg + Si	Versatile, formable, weldable, high strength, corrosion resistance. Mg + Si are forming Mg_2Si – Magnesium silicide in matrix.	Extrusion products for architecture Truck/marine frames (6061)
7xxx	Zn	Very high strength	Aircraft alloys (7050,7075)
8xxx	Li	Higher strength than 2xxx and 7xxx in aerospace uses. Low density, high hardness, excellent damage tolerance	Aircraft, aerospace alloys

In 1xxx series (pure Al) second digit indicates impurities, third and fourth digit express purity of Al equal to digits after decimal point. In 2xxx-8xxx alloys, second digit 0 means original alloy of Al + given element and 1-9 is indicating alloy modifications. Last two digits indicate different Al alloys in given group. [1]

Series 2xxx, 6xxx and 7xxx are suitable for heat treatment, based on heating the alloy, which homogenously distributes alloyed elements in the solid solution. After heating for dissolving alloyed elements in matrix, next step is quenching/rapid cooling, causing atoms to freeze in the solution. Then the atoms slowly combine into finely distributed stable precipitates, which increase mechanical properties. This process of letting atoms create finely distributed stable precipitates is called „aging“. Aging is a self-occurring process at room temperatures (natural aging) or it can be accelerated by using low temperature furnace (artificial aging). [3]

Series 3xxx, 4xxx and 5xxx are non-heat treatable. If there is need to strengthen them, cold-working needs to be used. This is done during rolling or forming into specific shapes. Cold-working is process of building dislocations and vacancies into the crystal structure of the material. This inhibits movement of atoms and causes higher strength. Some alloying elements improve this process, e.g. magnesium. [3]

1.2 Metallographic preparation of aluminium surface

1.2.1 Introduction to metallography

Metallography is one of the sub-fields of Materialography, specializing in preparing and examining structures of solid materials, in this case, metals. It is an investigative method, which enables us to examine microstructures and obtain its qualitative and quantitative properties. Other sub-fields of materialography are for example ceramography (ceramics), plastography (polymers), mineralogy (minerals) and others. Metallography can be described as a bridge between natural sciences and engineering. [5]

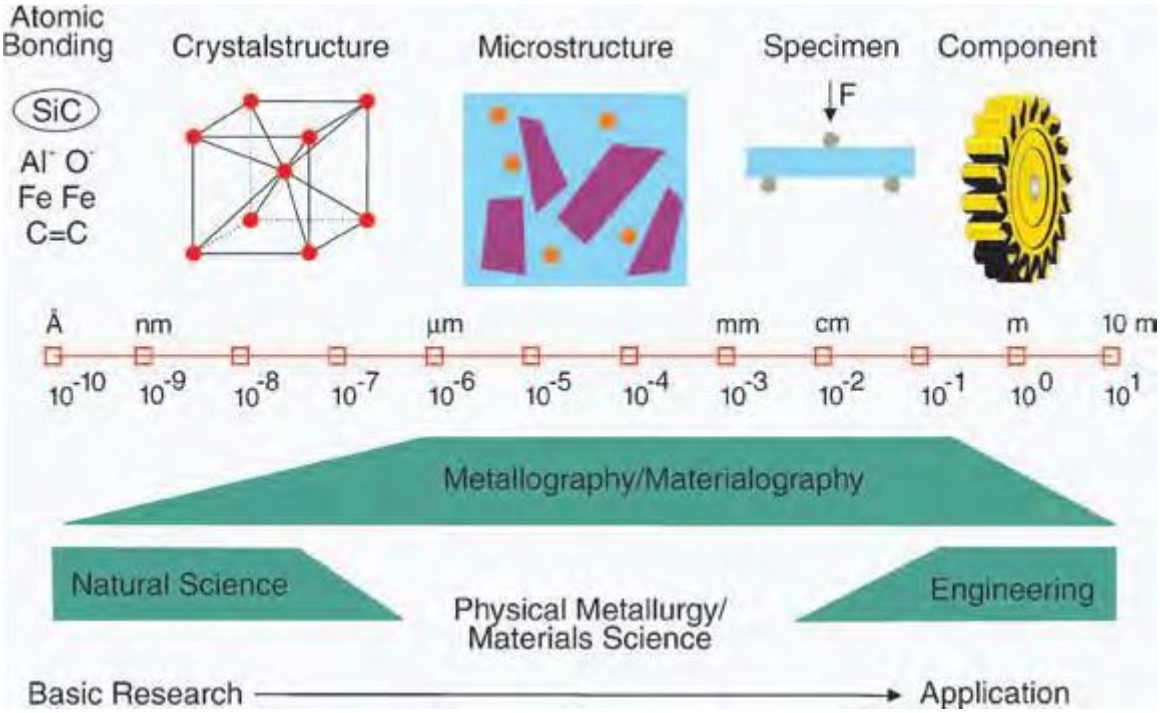


Fig. 2 - Bridge between natural sciences and engineering, metallography. [5]

In last years, there are more new materials than ever, so metallography and its knowledge expanded into incorporate material such as electronics, sporting goods and other every day-use things. Material properties, performance and reliability can be better understood through quality structure analysis. Metallography is therefore used in research of new materials, inspection, manufacture, production control and lastly, for failure analysis. [6]

Metallographic and Microstructural types of analysis:

Grain size, Porosity and voids, Phase analysis, Dendritic growth, Cracks and defects, Corrosion analysis, Intergranular attack (IGA), Coating thickness and integrity, Inclusion size/distribution/shape, Weld and heat affected zones (HAZ), Distribution/orientation of composite fillers, Graphite nodularity, Recast, Carburizing thickness, Decarburization, Nitriding thickness, Intergranular thickness, Intergranular fracturing, HAZ Sensitization, Flow-ine stress. [6]

1.2.2 Microstructure

Microstructure of a material is key to its properties (mechanical, electrical, magnetical, chemical) and for that reason, microstructure is studied deeply. Previous research in this field allows us to use known methods and apply them for new materials. Microstructure describes phases, ratio of phases, defects of phases, size, shape, arrangement. This information is different for each type of material and crystallic structures. It is also important to realize that their phases, grains and everything seen in microscopes are seen 2 Dimensional (2D), but in reality, they are 3 Dimensional (3D). [5]

Main goal of Metallographic specimen preparation is making the surface as fine and clean as possible.

True Microstructure [5]

Vilella and Samuels in their study defined term **„True Microstructure“,** which stands for „No deformation“, „No scratches“, „No pull-outs“, „No introduction of foreign elements“, „No smearing“, „No relief or rounding of edges“ [5]

„No deformation“ – plastically deformed layer should be insignificant or removed, if needed.

„No scratches“ – scratches give us information that surface is not ready yet, however, small scratches are tolerable.

„No pull-outs“ – In some materials, especially brittle ones, is risk of pulling particles out of surface, which leads to cavities and therefore corrosion.

„No introduction of foreign elements“ – abrasive grains should not be embedded into the surface during the grinding process.

„No smearing“ – In some materials -> their matrixes might smear themselves, which results in false perception of studied material.

„No relief or rounding of edges“ – two different constituents can develop a relief between them based on different characteristics (hardness for example). Retention of edges is also important in some cases.

Artifacts

Artifact = false structural detail introduced during the preparation. Artifacts are for example deformations, scratches, pull-outs and other phenomena stated in **True Microstructure** paragraph slightly above. The following phenomena can also be classified as artifacts: microcracks, comet tails, pitting, contamination and lapping tracks. [5]

1.2.3 Full process of specimen preparation

Foreword

Selecting the right preparation method is affecting the final prepared surface, creating artifacts during the process. Selecting optimal preparation method usually means choosing between mechanical or electrolytic polishing, sometimes chemical or mechanical-chemical polishing. In our case – when preparing Aluminium and its alloys – we will use mechanical polishing as the first big step and then trying various procedures and parameters during colloidal silica polishing and electrolytic polishing. Preparation consists of these steps: sectioning, mounting, grinding, polishing (diamond pastes, colloidal silicas, electropolishing) and in some cases etching. [2] [5]

Sectioning

Firstly comes choosing the correct section, to capture right structure to examine. Usually, more samples in various directions are taken – parallel to forming and perpendicular to forming. One of most important things in this step is cutting the sample without deforming it. Sample can't be mechanically or thermally altered by the process of cutting. The most commonly used method is abrasive cutting under cooling fluid. The use of thinner cutting wheels is usually the best way in order to avoid any thermal alteration of the sample. Large wheel diameters have lower chance of breaking. [2] [7]

Mounting

For smooth manipulation and for grinding in the grinding machine, samples are mounted into resin cylinders. This can be done by various methods – casting (sometimes vacuum casting) or combination of pressure and heat. The latter is used more, because it allows the user to mount samples more quickly – 15 minutes at the most, meanwhile cast resins solidify for hours before they can be used. Choice of the resin is between thermoplastics and thermosetting. Thermosetting resins are more used in general because of better edge retention and chemical indifference. Edge retention problem is solved by using compression mounted epoxy-based resins with filler, for example: DuroFast resin. For best results, choose low viscosity epoxy resin. [2] [6]

Grinding

Grinding can be done manually by hand or on automated devices. Most effective grinding abrasive for preparing Aluminium and its alloys is Silicon Carbide (SiC). In metallography, a series of steps is usually used– grinding wheels with finer and finer abrasive size – Preparing of aluminium wasn't much different in the past. In modern methods, only one abrasive size (therefore one grinding wheel) is used for grinding. This is possible by cutting the Aluminium sample with a wheel designed specifically for sectioning Aluminium. It is usually opted for 240-grit (P280) or 320-grit (P400). Water is usually used as coolant. Platen rotation is between 240 - 300rpm. Load – 22N (5lb). [2]

Polishing using Diamond pastes

Polishing is done by using diamond pastes as abrasive element and is folowed by final polishing using colloidal silica suspensions (specified later). In the past, MgO was used for final polishing, but that was difficult, results were poor and cloth-based polishing wheels were dirty and hard to clean. Because of its hardness, diamond is the best suited material for cutting through all materials. When using diamond as abrasive, either in suspension or paste, it is recommended to have the smallest chip size possible. Small chip size results in surface without artifacts and deformations. More resilient clothes are used in combination with small grain sizes like 3 μm, 1 μm or even 0,25 μm. Using resilient clothes, small grain size and low force will reduce chip size greatly. Similar method to „Polishing" is „Lapping". Differences between these two are that Lapping is done on hard plate tools, having coarser abrasives and resulting in rougher surface, whereas Polishing is using soft pads, finer abrasives and results in fine mirror-like surface. When lapping soft materials like Aluminium, there is a risk that abrasive particles will be pressed and embedded into surface accompanied by creation of deep deformations and hammering small particles out of specimen surface, which leads to lower quality of the final prepared surface. This means, Aluminium and other soft materials are not suited for lapping process, so diamond polishing comes into play for optimal preparation. [2] [8]

Polishing using Colloidal silicas

Colloidal silica is a suspension of fine SiO_2 particles in liquid phase. These particles are usually amorphous, nonporous and spherical. They are effective, because they provide dispersing action and chemical mechanical polishing simultaneously. Colloidal silicas are typically basic, with pH greater than 9,5. High pH values make colloidal silica almost „perfect“ suspension due to the electrochemical repulsive forces between fine silica particles. Up to 50 % of polishing solution is made of solid particles, while maintaining properties of suspension. Average particle size ranges between 0,02 – 0,07 μm [2][9]

Colloidal silicas have some problems, but they can be minimalized by these steps: Talking about sample, colloidal silica is harder to remove from surface than normal abrasives. To solve this, the surface is scrubbed with tuff or cotton soaked in soapy solution, then rinsed in water and dried in usual manner = water is replaced with ethanol and blown dry with warm air. Used cloth has to be rinsed well after using silica, so the small silica particles won't crystallize on surface of cloth and therefore won't ruin it. When using automated polisher, 20 s before end adding of colloidal silica stops, 10 s before end the water is turned on causing the colloidal silica to be flushed of the surface. Colloidal silica's with „non-freezing“ label, are minimizing crystalizing of particles and make the job easier. They produce similar results as normal silica suspensions. Revolver head of grinding machine can be also placed so that the outer edge of the head is running slightly over the edge of platen -> minimizing differential grinding effects and enhancing edge retention. [2] [9]

Based on the recommended processes, we will use OP-S and OP-U products by Struers in our experiments. Both OP-S and OP-U have particle size 0,04 μm. Talking exclusively about aluminium, higher pH of the silicas also enhances the polishing rate.

OP-S – Colloidal suspension especially suited for polishing of very ductile metals like aluminium, copper, tungsten, titanium, refractory metals or ceramics. It is suitable for mixing with chemical additives normally used for etching of the material. Mixing OP-S with acidic additives increases their chemical activity, shortening the final polishing time. OP-S itself has pH 9.8. Additives can be acidic (acetic acid), alkaline (potassium hydroxide), oxidation (hydrogen peroxide) or lubricating (glycerine). [10]

OP-U – Standard colloidal suspension best suitable for polishing of heterogeneous non-ferrous materials and composites. This suitability is due to the fact that OP-U polishes different phases evenly, resulting in planar specimen for good observation. It can be mixed with hydrogen peroxide or ammonia in limited quantities, but in general is not suited for mixing with additives. OP-U has also pH 9.8 but is less aggressive than OP-S suspension. [10]

Recommended processes

Below on tables 4, 5, 6 and 7 presented 4 recommended processes by literature and brochures of specialists.

Table 4 - Process 1: Van der Woort process [2]

Step	Grind. paper	Wat-er	Lubricant	Suspension	Load [N]	Time [s]	Platen speed [rpm]	Rota-tion
1	SiC 240 Grit	Yes	-	No	22	N/D	240-300	Co-rot.
2	MD-Dur	No	DP-Lubricant Green (Struers)	DP Paste-P 9 µm	22	300	120-150	Co-rot.
3	MD-Dac or MD-Sat	No	DP-Lubricant Green (Struers)	DP Paste-P 3 µm	22	180	120-150	Co-rot.
4 opti-onal	MD-Dac or MD-Sat	No	DP-Lubricant Green (Struers)	DP Paste-P 1 µm	22	120	120-150	Co-rot.
5	MD-Floc or MD-Chem	No	-	OP-S or OP-U	22	90-120	120-150	Cou-nter

Notes

Head speed were chosen by the rule that Platen and Head speed should differ minimally by 5 % and not differ more than 10 %. Therefore 5-10 % difference in those speeds depending on platen speed used. Load always round down, therefore 22 N -> 20 N in our case. At step 2, after 2 minutes start squirting 9µm DP Suspension P every 30 s. [2]

Table 5 - Process 2: Struers Aluminium Method 1 [11]

Step	Grind. paper	Wat-er	Lubricant	Suspension	Load [N]	Time [s]	Platen speed [rpm]	Rota-tion
1	SiC 320/500 Grit	Yes	-	-	25	Planar	300	N/D
2	SiC 800 Grit	Yes	-	-	25	30	300	N/D
3	SiC 1200 Grit	Yes	-	-	25	30	300	N/D
4	SiC 4000 Grit	Yes	-	-	25	30	300	N/D
5	MD-Mol (MD-Dac)	No	DiaPro MOL R3	DP Paste-P 3 μm	25	240	150	N/D
6	MD-Chem	No	-	OP-U or OP-S 0,04 μm	15	120-300	150	N/D

Notes

Consider rubbing SiC papers with wax to avoid coarse scratches.

Table 6 - Process 3: Struers Aluminium Method 2 (Alternative) [11]

Step	Grind. paper	Wat-er	Lubricant	Suspension	Load [N]	Time [s]	Platen speed [rpm]	Rota-tion
1	SiC 320 Grit	Yes	-	-	25	Planar	300	N/D
2	MD-Pan	-	DiaPro Pan 15	15 μm DIA	25	300	150	N/D
3	MD-Dac3 or MD-Dur3		DiaPro Dac3/Dur3	3 μm DIA	25	240	150	N/D
4	MD-Chem	No	-	OP-U or OP-S 0,04 μm	15	120-300	150	N/D

Notes

Consider rubbing SiC papers with wax to avoid coarse scratches.

Table 7 - Process 4: Struers Metalog Guide A [12]

Step	Grind. paper	Wat-er	Lubricant	Suspension	Load [N]	Time [s]	Platen speed [rpm]	Rota-tion
1	SiC 320 Grit	Yes	-	-	25	Planar /60	300	Co-rot.
2	MD-Largo	-	-	DiaPro Allegro/ Largo 9 μm	40	180	150	Co-rot.
3	MD-Mol	-	-	DiaPro Mol R 3 μm	25	300	150	Co-rot.
4	MD-Chem	-	-	OP-S NonDry, 0,25 μm	10	60	150	Coun ter-rot.

Notes

Consider rubbing SiC paper with wax to avoid coarse scratches.

Electropolishing

Electropolishing or „Electrolytic polishing“ is a method of specimen preparation based on electrolysis between specimen acting as anode and part of electropolishing machine acting as cathode. Mechanical grinding and polishing leaves a thin deformation layer because of its mechanical approach. Electropolishing is based on controlled dissolution of surface of the specimen, therefore leaving no layer of deformation. Electropolishing is an experimental method depending on many variables like choice and temperature of electrolyte, voltage, current density, time, choice of cathode material, spacing between anode-cathode and many others. Main advantages of this method are, for example preparation of metals with twinning dislocation mechanism or disturbed metal artifacts due to not touching polished surface. It can also reduce preparation time. Same electrolyte can be used for electrolytic etching, reducing voltage to 10 % of the value used for polishing. Disadvantages mainly concern electrolytes, some of them being poisonous, highly corrosive or explosive. This method is therefore best suited for pure metals and single phase alloys. When polishing multi-phase alloys, some problems occur such as difference in the polishing rates of phases: one phase is attacked preferentially before the other. Some electrolytes require not using plastic mounts and sometimes electropolished surfaces passivate and are difficult to etch afterwards. [13] [14]

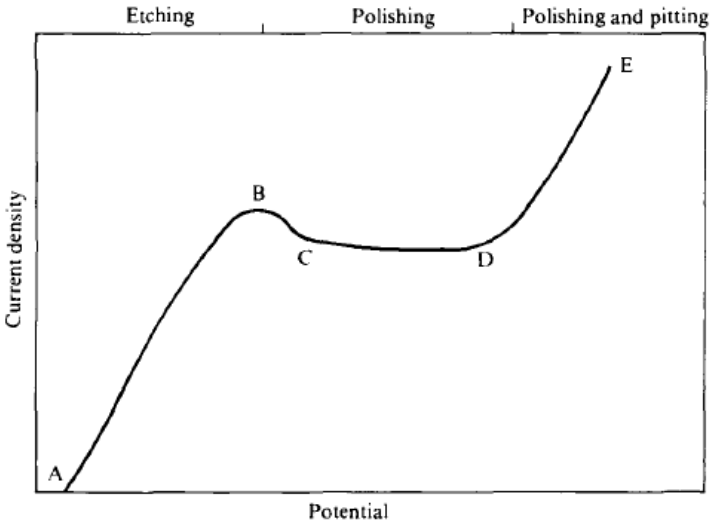


Fig. 3 - Voltage-Current density graph [13]

For best optimizing of electropolishing parameters, machines produce „Voltage-Current Density“ graphs seen in Fig. 3. Fig. 3 shows us how changing electropolishing parameters affects the electrolysis process. Low voltages (A-B) produce electrolytic etching, which can be sometimes our intention. Middle voltages (C-D) are ideal for electropolishing and therefore most used. High voltages (D-E) also polish, but they introduce pitting, which is not desired, so these high voltages are not used. [13]

Table 8 - Recommended electrolytes and parameters based on literature

Composition	**Voltage [V]**	**Temperature [°C]**	**Time [s]**	**Notes**
5 ml perchloric acid 95ml acetic acid [13]	25-60	Room temp.	120	Pregrind to 3/0 paper Stir in process Used for high purity Al
Struers A2 electrolyte [15]	25-39	22	15-20	All-round electrolyte for steel, aluminium, nickel, tin and titan

1.3 Microscopy

1.3.1 Optical microscopy

Optical or Light microscopy is done in light microscope, obtaining magnified picture using visible light, which is projected on human eye or photosensitive surface of imaging device. Light microscope contains two distinct sets of interlaced focal planes, 8 in all, between the illuminator and the eye. All of these focal planes play important role in formation of the image. Some of the planes are fixed, others are not. Their distance and position varies with focus position of the objective and illuminator. Simple principle of optical microscope is shown in fig. 4. [16]

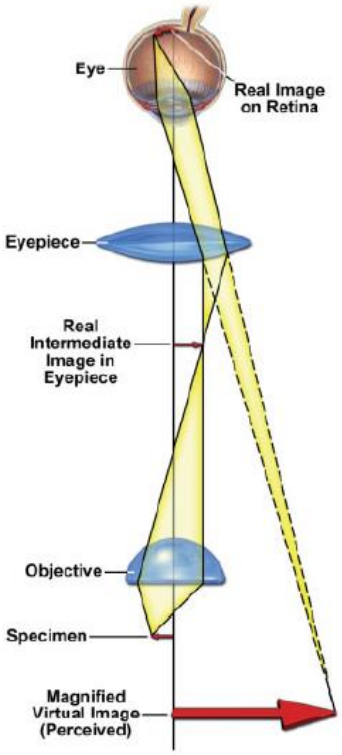


Fig. 4 - Schematic principle of human eye sight in light microscope

The commonly used method of illumination is „Koehler illumination“, giving us bright, uniform illumination and also positioning sets of images and diffraction planes at proper distances. For right illumination, a „Condenser“ is needed, which focuses light from the illuminator onto a small area on the specimen. Optical microscope has 2 main lenses -> objective and ocular, each having certain magnification. Magnification is the main parameter of light microscopes and is composed of 2 main features, objective magnification and ocular magnification. Therefore, Final magnification is magnification of objective multiplied with magnification of ocular, shown in equation 1. The objective collects light diffracted by the specimen and creates „Real image“, Ocular being lens near human eye, creating „Virtual image“. The interaction of light with surface of the specimen has many forms like absorption (making object opaque), transmission (making object transparent) reflection (making object reflected) or diffraction (making object scattering). [16]

$$M_{final} = M_{obj} * M_{oc} \, [-] \tag{1}$$

where:

M_{final} Final magnification

M_{obj} Magnification of objective

M_{oc} Magnification of ocular

Basic forms of optical microscopy examining are brightfield and darkfield. Brightfield is based on illuminating specimen from the above making the plane points reflect back to the above and deformed points reflect to the sides. Black deformed spots are therefore seen on the white plane. Darkfield is just the opposite. Illumination comes the from side, planar spots are reflected to the side and deformed spots are reflected to the lens. Fig.5 shows this concept [17]

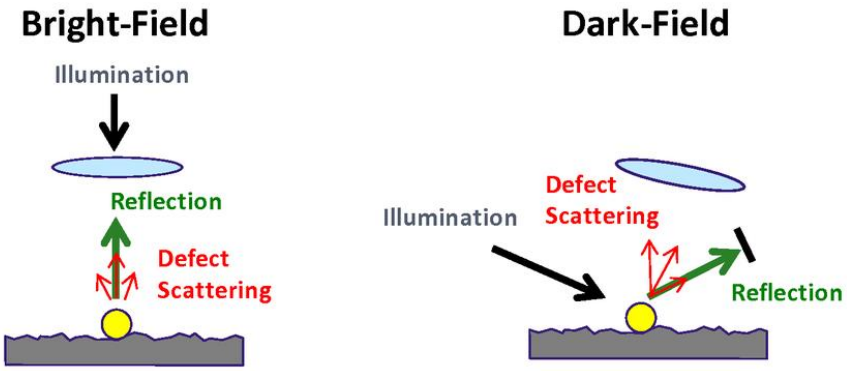


Fig. 5 – Principle of Bright-Field and Dark-Field forms of examining

1.3.2 Confocal (laser) microscopy

Confocal microscope is used in cases, when information outside the focal plane in light microscope negatively affects the measurement results. Confocal microscopy solves this problem by rejecting signals from nearby sources above and below the focal plane. This signal rejection is accomplished optically, by illuminating the specimen with a focused scanning laser beam and also placing the pinhole aperture in the image plane and in front of electronic photon detector. Another method, how to produce confocal image is using a spinning „Nipkow“ disk, which gives us image using literally thousands of scanning beams in tandem. At the Z-axis of the microscope is a stepper motor that changes the working distance and therefore changes the focus of the microscope by small steps up and down. This enables us to acquire a set of images on different levels in Z-axis, which results in making a 3D view of the specimen surface in small dimensions. These 3D images are not as complex and as deep as 3D images from electron microscopy, but they can still offer a lot of information, for example using 3D data to measure Surface roughness of the specimen. [16]

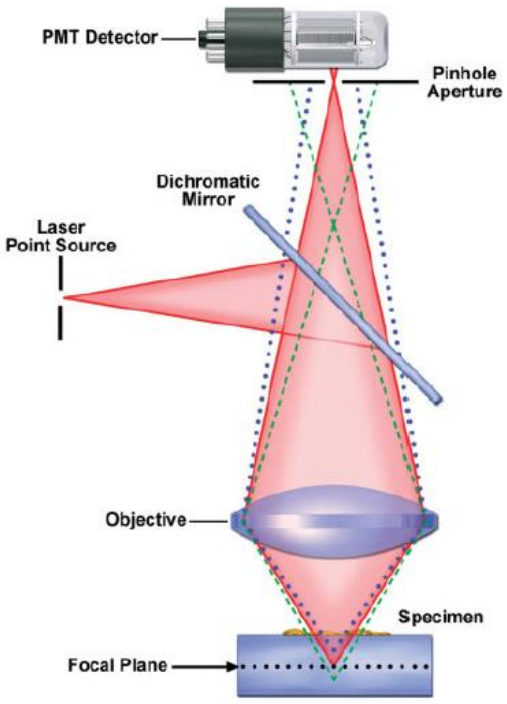


Fig. 6 – Schematic principle of confocal microscope

Wavelengths are excited from laser point source onto the surface on certain scanning point on the specimen. Wavelengths emitted from confocal point in the specimen are seen on the image, because they have plane confocal with pinhole aperture. Fluorescent light emitted at points above and below has no confocal plane with pinhole aperture and therefore isn't coming through and is not shown on the produced image. This principle is shown in fig. 6. [16]

1.3.3 Electron microscopy (SEM)

SEM – Scanning Electron Microscope is an instrument, which creates magnified images revealing microscopic-scale information about crystallographic, physical and chemical properties of observed specimen. The principle of SEM is creation of finely focused beam of energetic electrons by means of emission from an electron source. Energy of the electrons in the beam is one of the parameters selected by person operating the microscope. Energy E(0) is typically chosen in the range of 0,1 to 30 eV. Electron is emitted from the source, accelerated to high energy and comes through the series of apertures, magnetic/electrostatic lenses and electromagnetic coils as shown in fig 7. [18].

This sequence of steps is reducing the beam diameter and allowing it to scan the surface of the specimen in raster format. Raster scanning allows us to have discrete information about every point in picture. [18] [19]

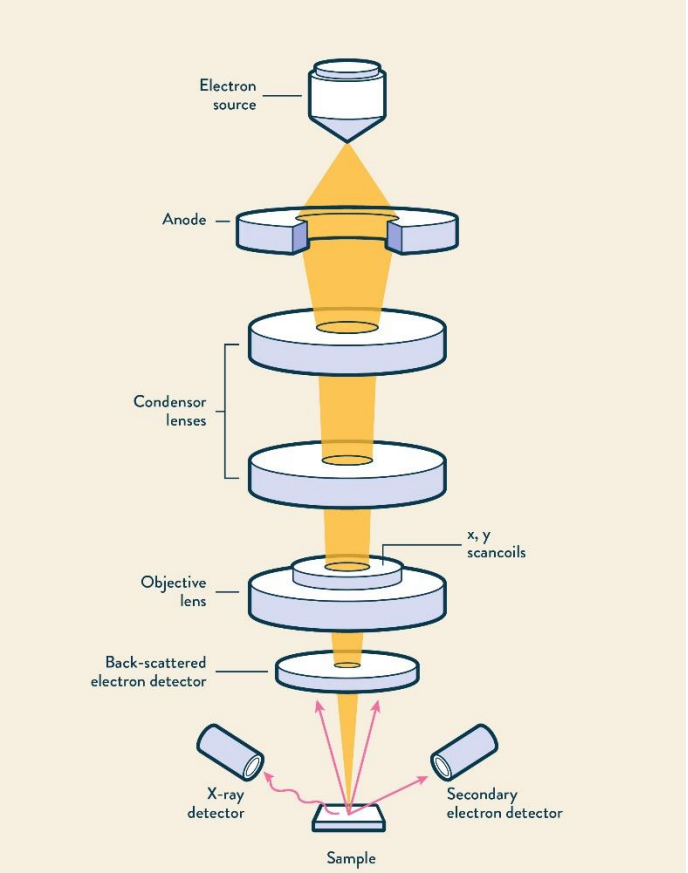


Fig. 7 – Scheme of SEM tube with source, lenses, coils, detectors and surface

After interaction of the electron beam and the specimen, each of these discrete locations provide 2 outgoing electron products – Backscattered electrons (BSE) and Secondary electrons (SE). Shown in fig. 8 [19]

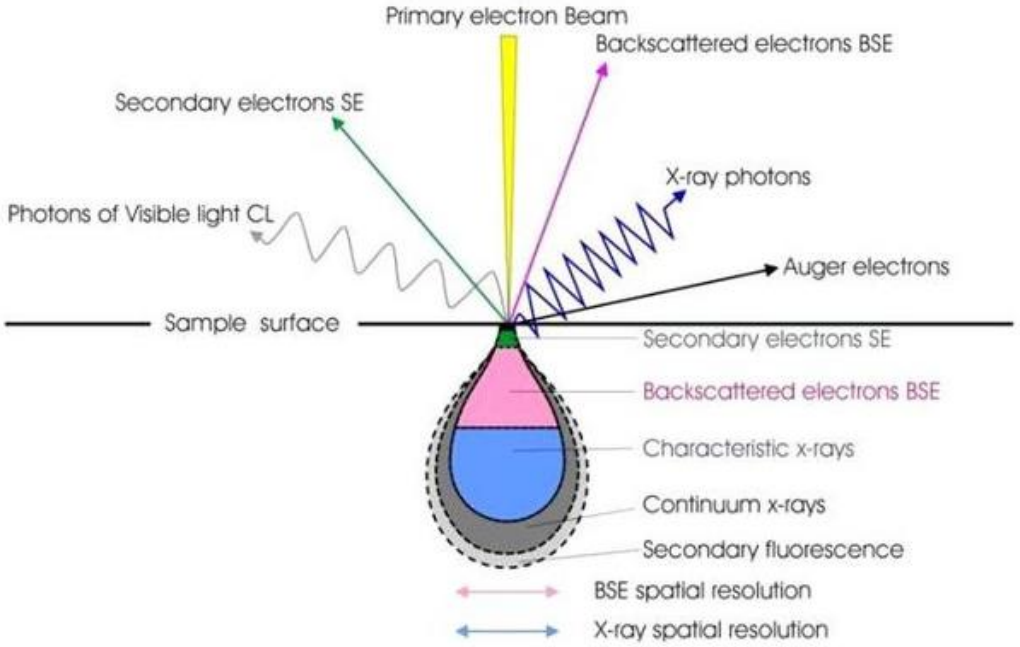


Fig. 8 – Electron beam interaction with surface of the specimen

BSE are bounced beam electrons that emerge from the specimen with large fraction of incident energy, caused by scattering and deflection of electric fields of atoms in the specimen. [20]

SE are electrons of the specimen, which escape the specimen surface after beam electrons ejected them from atoms on the surface. [20]

Incoming beam electrons have high energy, but they and secondary electrons experience low kinetic energy transfer, meaning that secondary electrons escape the surface with very low kinetic energies, generally 0-50 eV, usually lower than 5 eV. These outgoing electron signals are measured using electron detectors. Prime example of those detectors is for example „Everhart-Thornley secondary electron detector“, which is sensitive to both BSEs and SEs or „Dedicated backscattered electron detector“, which is sensitive only to BSEs and insensitive for SEs. Each detector at each individual raster scan location is digitized and saved into the computer memory. Those informations are seen by variation of „Grey-level“, at the given location of single point. Grid made of those points makes an image, which enables us to see and examine the specimen. The chamber of the conventional SEM microscope must operate in high vacuum condition, under 10^-4 Pa) to minimize encounters of atoms and molecules of gases in the air with beam electrons (and also BSEs and SEs) and ensuring that the beam can hit the surface correctly. The specimen needs proper grounding, usually done with conductive coating to provide an electrical discharge path. Otherwise, the insulated specimen would develop a surface electrical charge because of beam electrons interaction with surface, alternating the result and impact the measurement. [20]

SEM electron parameters are optimized based on the information the user wants to obtain. For high spatial resolution imaging and good scale detail a small beam diameter is chosen. This, however, has also downsides, like reduction of the beam current, which leads to low contrast.

Inversely, choosing high beam current, improves the visibility of low contrast objects. There is always threshold contrast, below, which the features of the specimen won't be visible. This threshold contrast depending on relative size and shape of the feature of interest, meaning that, under threshold contrast, the large objects will be visible, whereas small objects will not. [20]

Threshold contrast is lowered by 3 things: Increasing the beam current, pixel dwell time or detector efficiency. Increasing beam current leads to large beam size, causing the resolution to fade. There is a need to find a compromise between resolution and visibility, leading to limitations. To increase the depth of the field, the beam divergence angle can be minimized. This is achieved by the appropriate aperture selection and optimization of the working distance (specimen-to-objective lens distance). These parameters can be effectively optimized to achieve small beam convergence angles, good focus and depth of the field. [20]

SEM images can give us these informations: [20]

a) Compositional microstructure – differences between atomic number (Z) of certain rastered points in the specimen.
b) Topography (Shape) – topographic information is measured using local surface inclinations as small as few degrees. Spatial resolution ranging from incident beam diameter 1nm or less to 10 nm or more, varying with material and geometry of the edges.
c) Visualization of third dimension – with large depth in field, 3D structure of the specimen surface can be visualized. In conventional X-Y image, the 3D information is projected on 2D image, leading to foreshortening. This can be fixed by using stereomicroscopy, creating pictures by combining 2 independent views with small angular differences.
d) Other properties such as crystal structure, grain boundaries, crystal defects, crystal deformation effects, magnetic microstructure, magnetic domains and interfaces, applied electrical fields in microstructures, cathodoluminescence.

Strengths and weaknesses of SEM [21]

Strengths: variety of specimen to be examined, user friendly and automated equipment, fast measurement and analysis times, large depth of field, microchemical analysis of elements from Be to U, using dry or wet samples, nondestructive, high spatial resolution, capability of several modes (imaging, spectroscopy, diffraction analysis).

Weaknesses: limited specimen size, only solid samples, need of vacuum in the chamber (cost and vacuuming times), large space requirement for instrument (5x5m), non-conductive specimen need to be coated.

EDS – Electron Dispersive X-Ray Spectroscopy

EDS is giving us information about elemental composition of given specimen. Principle of this analysis is that when electron beam interacts with specimen, it produces X-ray photon emissions. There are specifically 2 types of X-ray photon emissions: [20]

- Characteristic X-rays with specific energy for each specific element (except H and He, which do not emit X-rays) giving us information what the specimen matrix is made of.
- Continuum X-rays, occurring at all photon energies, from measurement threshold to E(0), forming background under the characteristic X-rays.

Obtained X-rays characteristics are used for identifying and quantifying specific elements in the area of volume covered by electron beam. [20]

Depending on the mass fraction (C) of the constituent, it is being referred to as: [20]

$0{,}1 < C \leq 1$ – „Major Constituent“

$0{,}01 < C < 0{,}1$ „Minor Constituent“

$C < 0{,}01$ „Trace Constituent“

X-ray spectrum is measured using semi-conductor energy dispersive X-ray spectrometer, so called „EDS“, which can detect photons from threshold of approx. 40eV up to E(0) (as much as 30 000eV). When qualitative analyzing X-ray spectrum, user encounters many challenges. Some element peaks interfere with each other, e.g. Ti and Ba; S/Mo and Pb. These interferences occur especially between major, minor and trace constituents. Skilled person is needed to trace these false assumptions and interferences and delete them. The next step after qualitative analysis is quantitative analysis of X-ray spectrum. Intensity for each peak is automatically determined by peak fitting procedures, e.g. multiple linear least squares method. [20]

2 Specimens and Instruments

2.1 Specimens

Chemical composition of used specimens is 99,5 % aluminium and 0,5 % of elements present in the form of precipitates. To verify the purity of aluminium matrix and get more information about precipitates, we used EDS analysis.

Fig. 9 shows choice of EDS spots to measure purity of the matrix and composition of precipitates. In fig. 10 we can see EDS spectrum of spot 1, showing us that matrix is clearly made out of aluminium.

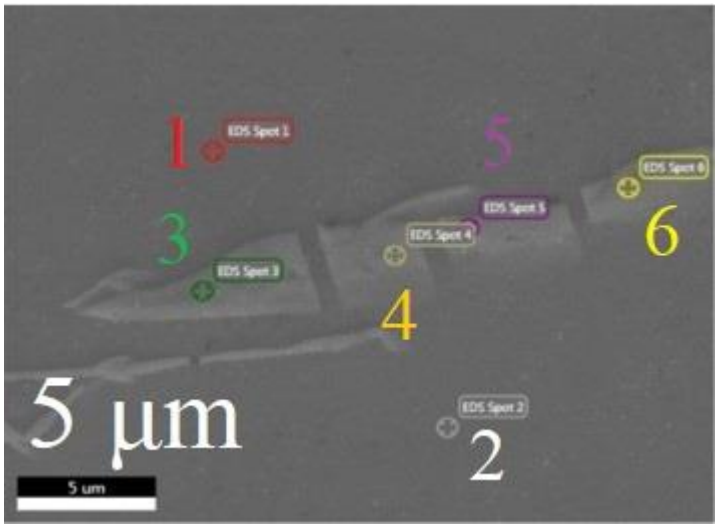


Fig. 9 - EDS spots for verification of Al purity and observation of precipitates

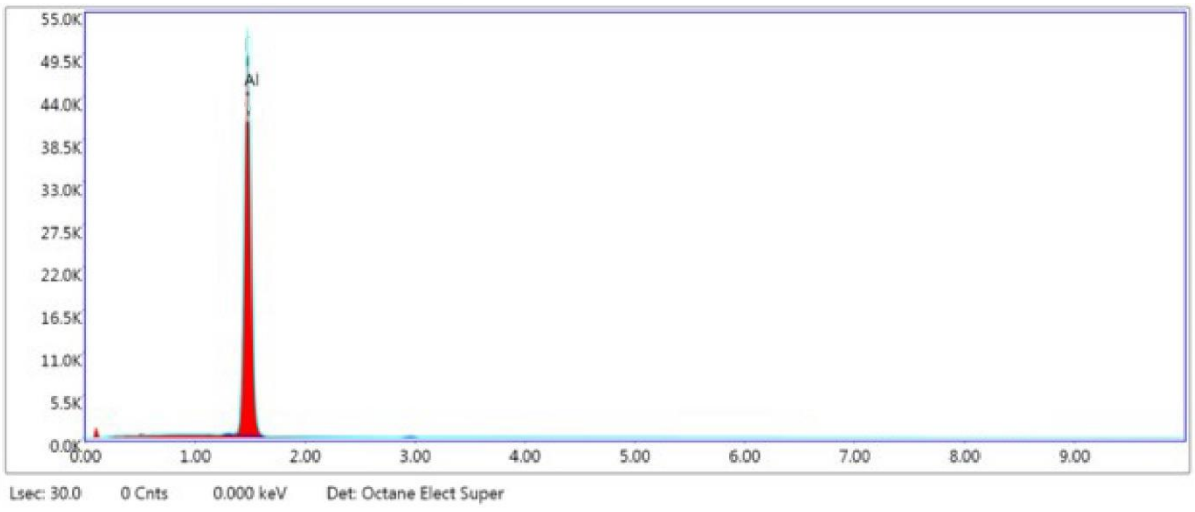


Fig. 10 - Verification of purity of Aluminium matrix of EDS spot 1

In figure 11 we can see EDS analysis of spot 3, which reveals that precipitate consists mainly of Aluminium, Iron and Silicium.

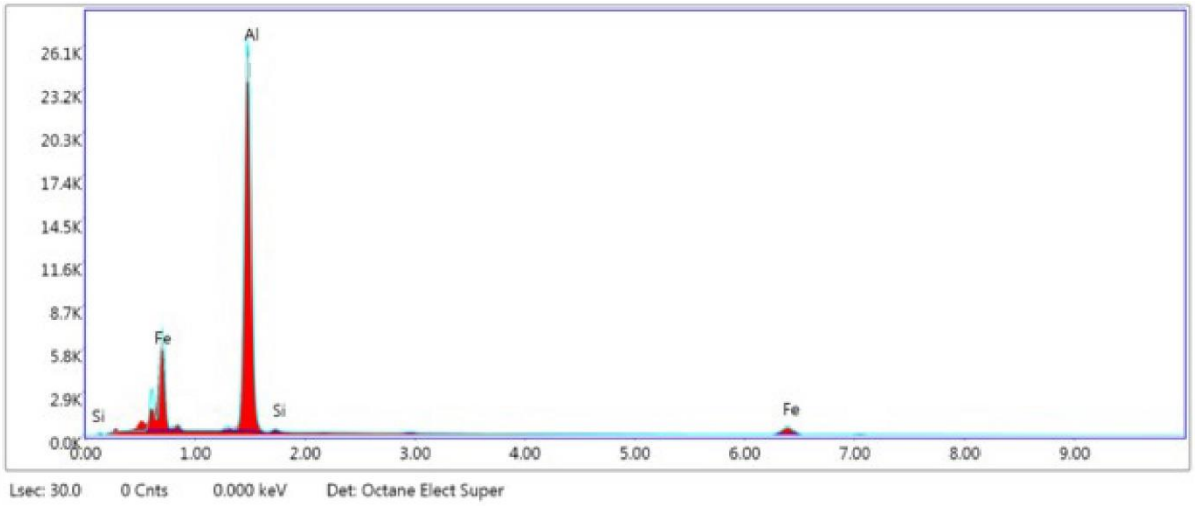


Fig. 11 - β-AlFeSi precipitate EDS spectrum of spot 3

Iron probably got into the used specimens during the melting process from the walls of bucket. In aluminium (and generally Al-Si alloys), iron is considered an impurity. Iron has almost no solubility in aluminium and its alloys. This means that even very small volumes of iron create intermetallic phases. Two most important intermetallic phases of iron in aluminium are:

Al_5FeSi being referred to as „β-AlFeSi“, which is characterized as pins in material.

$Al_{15}(Fe,Mn)_3Si_2$ being referred to as „α-AlFeSi“, „Chinese writing“ or „Fish bone“ , being more compact than β-AlFeSi.

Table 9 shows weight and atomic percentages of aluminium, iron and silicium in precipitates based on EDS measurements of spots 3, 4, 5 and 6.

Table 9 - Al-Fe-Si weight and atomic percentage in precipitate

Spot	Al wt%	Fe wt%	Si wt%	Al at.%	Fe at.%	Si at.%
3	60.75	21.02	18.23	77,91	13.03	9.06
4	57.55	21.37	21.08	75,65	13.57	10.78
5	62.68	16.28	21.04	79.72	10.00	10.28
6	62.13	18.29	19.58	79.04	11.24	9.72

These Al-Fe-Si ratios in table 9 and absence of manganum show that our specimen have Al_5FeSi precipitates, not $Al_{15}(Fe,Mn)_3Si_2$. We will mainly focus on β-AlFeSi in this thesis.

β-AlFeSi is created during solidification process at approximately 580 °C, therefore before exclusion of Silicium lamellas. It has plate-like shape (looking like pin under the microscope) with monoclinic lattice. β-AlFeSi plates disrupt the structure of pure Al and therefore lead to significant aggravation of mechanical properties. β-AlFeSi also has far worse effect on mechanical properties than compact α-AlFeSi. Basic casted aluminium product is supposed to have Fe(max) = 0,6 wt%, for high-strength casting Fe(max) = 0,15 wt%. When concentration of Iron exceeds 0,4 wt%, it is recommended to add manganum, which enables the given material to create more compact α-AlFeSi phase instead of β-AlFeSi. Considering machinability of Al with Fe intermetallics, when the volume of Fe is lower than 1,2 wt%, it has no effect on machinability. Otherwise, the machinability starts to decline. Fe in Al alloys also slightly lowers corrosion resistance. We therefore try to keep Fe at minimum volumes in alloys used in corrosive environments. [22]

2.2 Metallographic equipment

For cutting the specimens and resins, we used Secotom 60 cutting machine by Struers. It has wide variety of clamps to secure the specimen for proper cutting, many different cutting wheels with hardnesses according to cut material and best safety measures in case of wheel cracking accident. Shown in fig. 12 with specification available at: [23]

Fig. 12 - Secotom 60 cutting machine [23]

For hot mounting specimens into resin, CitoPress-5 by Struers was used. It is suitable for many hot-mounting resins with process times ranging from 5-15 minutes, making it a very effective tool for automatic preparation of resin cylinders. Shown in fig. 13 with specification available at: [24]

Fig. 13 - Citopress 5 mounting machine [24]

Planar grinding, fine grinding, polishing using diamond pastes and colloidal silicas polishing operations were done on Tegramin-20 by Struers. Grinding/polishing discs are held by magnetic rotating wheel. The machine puts pressure on specimen directly (not on the whole specimen holder) and can be set to specific parameters. Metallographic preparation is therefore better and more repeatable than manual preparation. It can be seen in fig 14. with specification available at: [25]

Fig. 14 - Tegramin-20 polishing machine [25]

Electropolishing of specimens was performed on Lectropol-5 by Struers. Apart from choosing specific parameters, the machine is capable of scanning current density and other parameters for best results. It can be also used for electrolytic etching and offers database of processes for optimal laboratory use. Lectropol-5 is shown in fig. 15, with specification available at: [26]

Fig. 15 - Lectropol-5 electropolishing machine [26]

2.3 Microscopes

After metallographic preparation on these machines, we moved to microscopy analysis.

Optical microscopy experiments were conducted on Zeiss Axio Observer 7 materials. This microscope features motorized focus in Z-axis, automatic component recognition, touchscreen display and a remote control option. It is ideal for quick observation in laboratory conditions when quickly changing specimens or objectives but gives solid and reliable results. Shown in Fig 16. Parameters and specifications available at: [27]

Fig. 16 - Axio Observer 7 materials [27]

Confocal microscope used for surface roughness measurements was Violet semiconductor laser-VK-X1100. It offers total magnification up to 28 800x, having wavelength of 404nm and when measuring height, 0,5nm display resolution. VK-X1100 Head is shown in fig.17. More parameters and specifications available at: [28]

Fig. 17 - VK-X1100 Head [28]

For electron microscopy purposes, such as measuring at high magnifications and EDS analysis, we use the Magellan 400 by Thermo Fisher Scientific. Magellan 400 has sub-nanometer resolution in voltage range of 1-30 kV, making it suitable for examining nanomaterials and nanostructures like multi-layered coatings or other nanotechnological processes. This microscope can also examine non-conductive specimens. It has STEM, SE, BSE detectors for detection of all kinds of electrons. Apart from EDS analysis, it is also suitable for EBSD analysis for grain orientation and CL – Cathodoluminescence images. Magellan 400 and detail of the vacuum chamber are shown in figures 18 and 19. [29] [30]

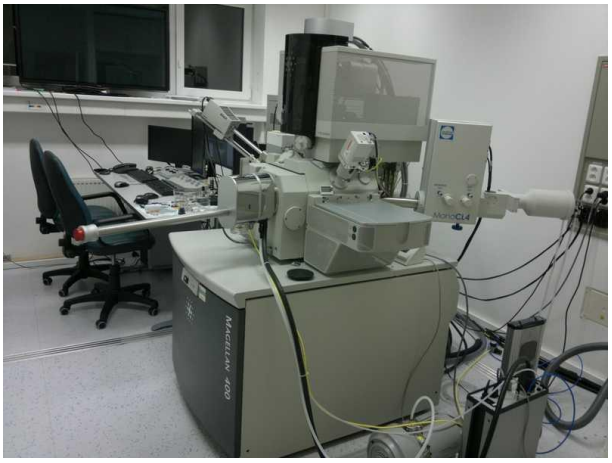


Fig. 18 - Magellan 400 setup [29]

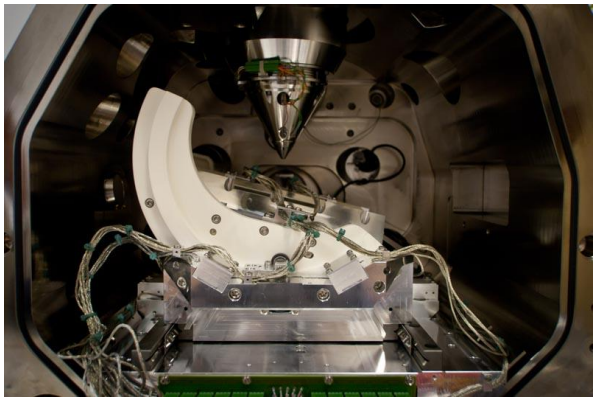


Fig. 19 - Magellan 400 - Specimen chamber [30]

3 Experiments, Results

3.1 Motivation

Main goal of the thesis is to optimize the conventional metallographic preparation processes recommended for pure aluminium in order to obtain the best surface quality suitable for specific needs of advanced scanning electron microscopy. Advanced SEM techniques are highly sensitive to surface quality due to extreme shallow interaction volume at low landing energies of the primary beam. Thus, standard surface preparation techniques used for the optical microscopy become insufficient for these purposes. Metallographic specimen preparation of pure aluminium is challenging, due to its low hardness. We therefore need to choose parameters very carefully. The aim of the thesis is to optimize the standard metallographic techniques used for Al and development of advanced methods for pure aluminium specimens in order to acquire the best quality surface for the low voltage SEM. The best metallographic technique based on mechanical, mechanical-chemical (colloidal silicas) and electro-polishing technique will be proposed for pure aluminium.

3.2 Mechanical polishing

For simplification, used processes were given abbreviations on table 10:

Table 10 - Mechanical processes abbreviations

VDW	Modified Process 1: Van der Woort process
MAIN	Modified Process 2: Struers Aluminium Method 1
ALT	Modified Process 3: Struers Aluminium Method 2 (Alternative)
MTA	Modified Process 4: Struers Metalog Guide A

Firstly, specimens were prepared just mechanically, based on recommended processes from chapter 1.2.3 (page 7). Specific used parameters are shown down below on tables 11, 12, 13 and 14.

Table 11 - Modified Process 1: Van der Woort process

Step	Grind. paper	Water	Lubricant	Suspension	Load [N]	Time [s]	Platen speed [rpm]	Head speed [rpm]	Rotation
1	SiC 240 Grit	Yes	-	-	15	120	240	150	Co-rot.
2	MD-Pan	No	-	DiaDuo 9 µm	10	180	150	100	Co-rot.
3	MD-Dac	No	IPA	DP Paste-P 3 µm	10	180	150	100	Co-rot.
4	MD-Sat	No	IPA	DP Paste-P 1 µm	5	240	150	100	Co-rot.
5	MD-Sat	No	IPA	DP Paste-P 0,25 µm	5	240	150	100	Co-rot.

Table 12 - Modified Process 2: Struers Aluminium Method 1

Step	Grind. paper	Water	Lubricant	Suspension	Load [N]	Time [s]	Platen speed [rpm]	Head speed [rpm]	Rotation
1	SiC 220 Grit	Yes	-	-	15	60	300	150	Co-rot.
2	SiC 500 Grit	Yes	-	-	10	30	300	150	Co-rot.
3	SiC 1200 Grit	Yes	-	-	10	30	300	150	Co-rot.
4	SiC 4000 Grit	Yes	-	-	5	60	300	150	Co-rot.
5	MD-Mol	No	IPA	DP Paste-P 3 µm	5	240	150	100	Co-rot.
6	MD-Dac	No	IPA	DP Paste-P 1 µm	5	240	150	100	Co-rot.
7	MD-Dac	No	IPA	DP Paste-P 0,25 µm	5	240	150	100	Co-rot.

Table 13 - Modified Process 3: Struers Aluminium Method 2 (Alternative)

Step	Grind. paper	Water	Lubricant	Suspension	Load [N]	Time [s]	Platen speed [rpm]	Head speed [rpm]	Rotation
1	SiC 220 Grit	Yes	-	-	15	60	300	150	Co-rot.
2	SiC 500 Grit	Yes	-	-	15	60	300	150	Co-rot.
3	SiC 1200 Grit	Yes	-	-	10	120	300	150	Co-rot.
4	MD-Pan	-	-	DiaDuo 9 µm	10	300	150	100	Co-rot.
5	MD-Mol	No	IPA	DP Paste P 3 µm	5	240	150	100	Co-rot.
6	MD-Dac	-	IPA	DP Paste-P 1 µm	5	240	150	100	Co-rot.
7	MD-Dac	-	IPA	DP Paste-P 0,25 µm	5	240	200	100	Co-rot.

Table 14 - Modified Process 4: Struers Metalog Guide A

Step	Grind. paper	Water	Lubricant	Suspension	Load [N]	Time [s]	Platen speed [rpm]	Head speed [rpm]	Rotation
1	SiC 220 Grit	Yes	-	-	20	60	300	150	Co-rot.
2	MD-Largo	-	-	DiaDuo 9 µm	10	180	150	100	Co-rot.
3	MD-Mol	-	IPA	DP Paste P 3 µm	5	240	200	100	Co-rot.
4	MD-Dac	-	IPA	DP Paste-P 1 µm	5	120	200	100	Co-rot.
5	MD-Dac	-	IPA	DP Paste-P 0,25 µm	5	120	200	100	Co-rot.

Final mechanical polishing step was grinding with 0,25 μm diamond paste, which was supposed to produce high quality surface without artifacts**.** However, when we look at surface of all prepared samples just by naked eye, we see highly scratched surface revealing nothing, therefore not ready for electron microscopy. Nevertheless, we chose to examine it in optical microscope Zeiss Axio Observer 7 materials (specified in chapter 2.3). The scratched surface of process MTA (Struers Metalog Guide A) is shown in figures 20 and 21, with all other specimens showing the same scratches. These scratches are probably present because low strength and hardness of pure aluminium. This is showing us the importance of using not only mechanical polishing, but also other preparation methods like colloidal silicas, electropolishing, chemical etching, etc.

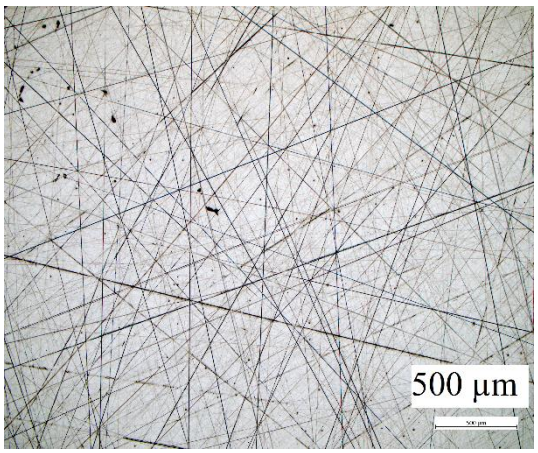


Fig. 20 - 50x Magnification of scratched surface prepared by MTA process with last step 0,25 dia paste

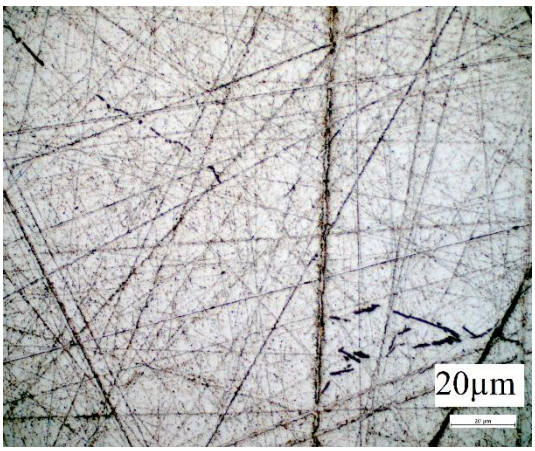


Fig. 21 - 1000x Magnification of scratched surface prepared by MTA process with last step 0,25 dia paste

3.3 Colloidal silicas polishing

Firstly, specimens were prepared by mechanical polishing processes specified above in chapter 3.2 – Mechanical preparation and as the final step, polishing using colloidal silicas was added. Used parameters are shown in table 15. Choice of colloidal suspensions was added based on sources same as before mentioned mechanical processes. OP-S and OP-U suspensions and their differences are specified in chapter 1.2.3 (page 6).

Table 15 - Parameters of colloidal silica polishing used for experiments

Process	Grinding paper	Water /Lubri -cant	Suspension	Load [N]	Time [s]	Platen speed [rpm]	Head speed [rpm]	Rotation
VDW [2]	MD-Floc	No	OP-U	5	180	200	100	Counter -rot.
MAIN [11]	MD-Chem	No	70% OP-S 30% H2O	5	180	200	150	Co-rot.
ALT [11]	MD-Chem	No	OP-U	5	180	150	100	Counter -rot.
MTA [12]	MD-Chem	No	70% OP-S 30 %H2O	5	180	150	100	Counter -rot.

After using colloidal silica suspension for just 3 minutes, specimens were without scratches seen by naked eye and finally ready to examine. They were first examined in Optical Microscope with magnification 50x shown in figures 22-25. We can see that specimens prepared by VDW and ALT processes are underetched. These processes were prepared by the same silica suspension OP-U. Processes MAIN and MTA prepared by OP-S suspension are evidently better etched and good looking in optical microscope. This comparison shows that OP-S suspension is more aggressive than OP-U when polishing pure aluminium. This measurement gave us first insight on the surface of pure aluminium and confirmed that the specimens are ready for examination under the electron microscope.

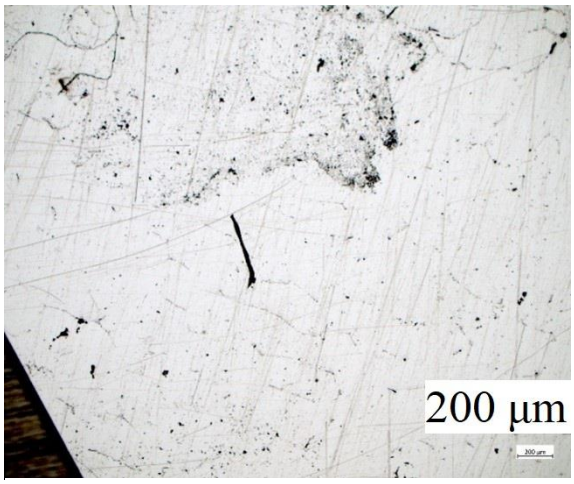


Fig. 22 - VDW 50x, OP-U 3 min

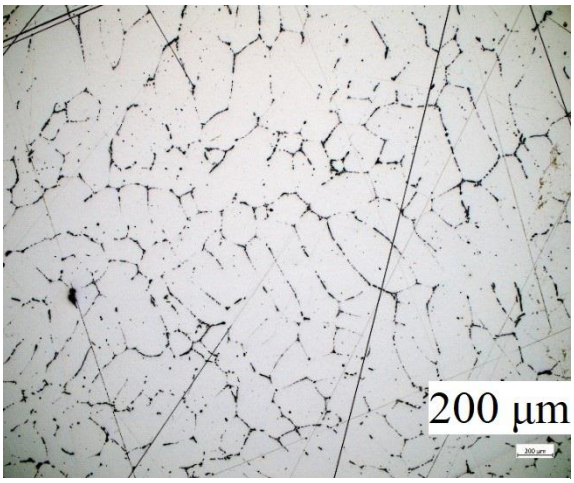


Fig. 23 - MAIN 50x, OP-S 3 min

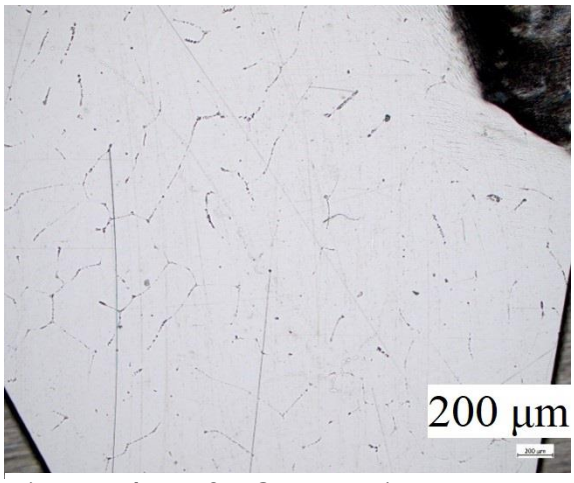


Fig. 24 - ALT 50x, OP-U 3 min

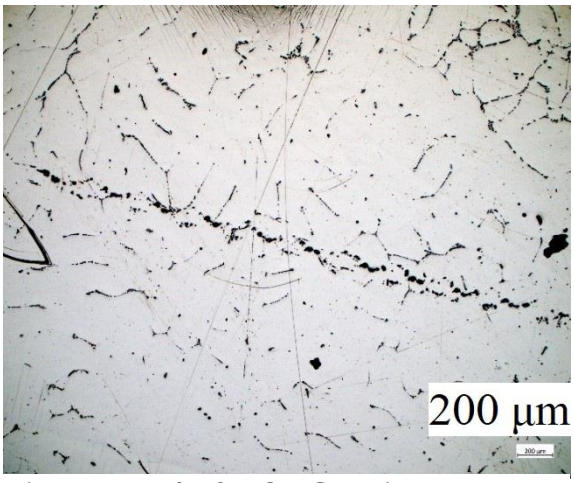


Fig. 25 - MTA 50x, OP-S 3 min

Specimens were moved and examined in electron microscope Magellan 400 (specified in chapter 2.3). Measured parameters are specified below for magnifications 1000x and 5000x and also measured images of specimens.

Table 16 - 1000x magnification measurement parameters

HFW	HV	Current	Detector	Mode	Magnification
149 µm	1.00 kV	0.10 nA	ETD	SE	1000x

1000x magnification process comparison:

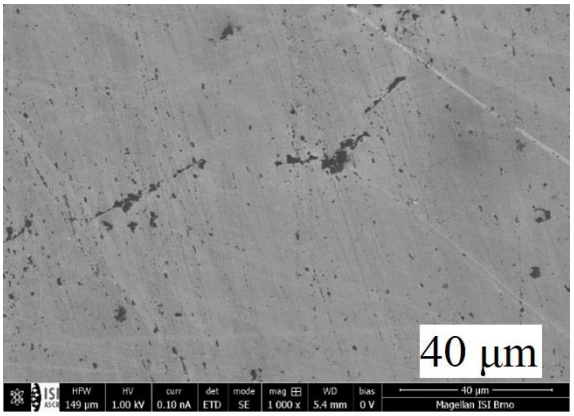


Fig. 26 - VDW 1000x, OP-U 3 min

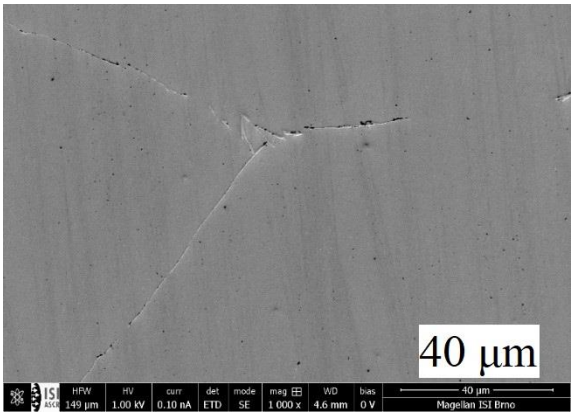


Fig. 27 - MAIN 1000x, OP-S 3 min

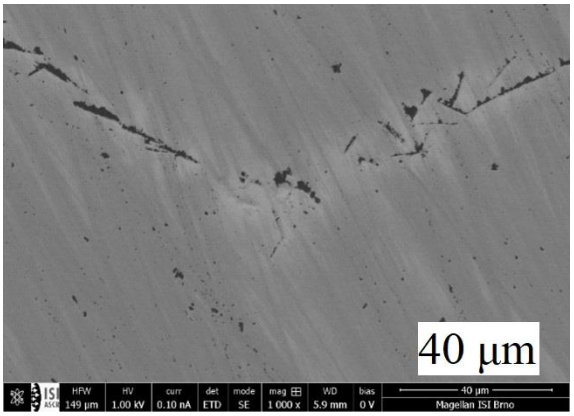


Fig. 28 - ALT 1000x, OP-U 3 min

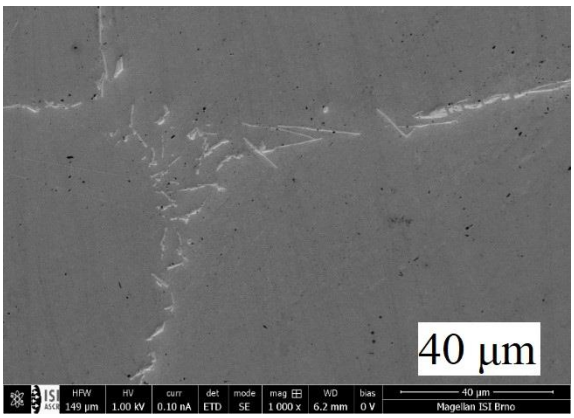


Fig. 29 - MTA 1000x, OP-S 3 min

Table 17 - 5000x magnification measurement parameters

HFW	HV	Current	Detector	Mode	Magnification
29.8 µm	1.00 kV	0.10 nA	ETD	SE	5000x

5000x magnification process comparison:

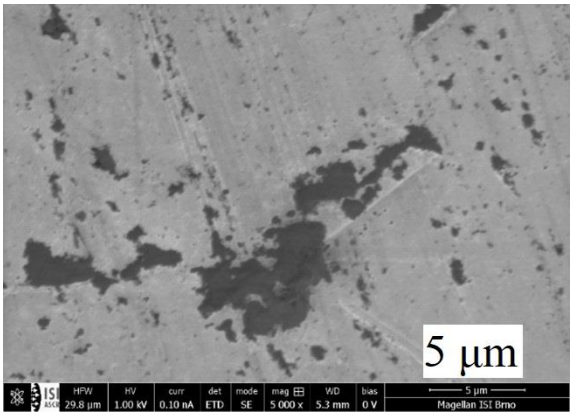


Fig. 30 - VDW 5000x, OP-U 3 min

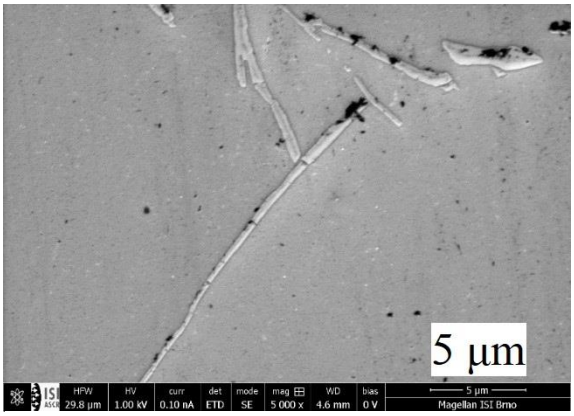


Fig. 31 - MAIN 5000x, OP-S 3 min

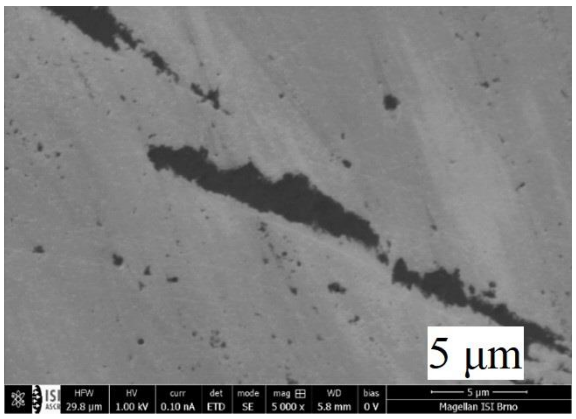


Fig. 32 - ALT 5000x, OP-U 3 min

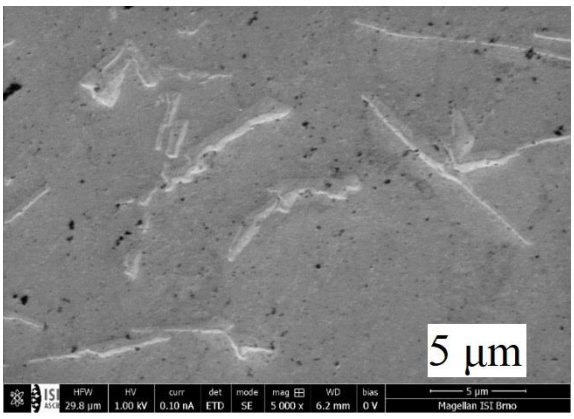


Fig. 33 – MTA 5000x, OP-S 3 min

We can see that specimens prepared by Main process and Metalog A process (Figures 27, 29, 31, 33) are clearly better prepared than Van der Woort and Alternative process as it seemed during optical microscopy (Figures 26, 28, 30, 32).

On images of specimens prepared by VDW and ALT processes can be seen black stains. These stains are result of contamination, which was caused by the OP-U suspension. OP-U suspension reacted with the surface and contaminated it. This contamination degraded the quality of the prepared surface. OP-U suspension is therefore not good for preparing pure aluminium.

On the other hand, processes MAIN and MTA with OP-S suspension as last polishing step look very good in SEM. These images allow us to see clean aluminium matrix and also β-AlFeSi precipitates (specified in chapter 2.1) for the first time. OP-S proved to be a very good choice of colloidal suspension and we will continue using it in our next experiments.

After finding out that OP-S is better for our cause than OP-U, we have to decide, which of the 2 processes using OP-S is better.

MAIN and MTA processes share a lot of similarities, especially in the final diamond paste polishing steps. Biggest difference lies in the middle set of steps. MAIN process uses grinding with consecutive SiC wheels with grits 500, 1200 and 4000 before moving onto diamond paste polishing. MTA process instead of SiC wheels uses one MD-Largo wheel with DiaDuo 9 μm suspension. To decide, which process is more suitable for the best preparation possible, we made new specimens using the same processes, but stopping preparation after SiC4000 Grit and MD-Largo steps to see the difference in surface roughnesses.

Those „partially prepared“ specimens were examined in Violet VK-X1100 Confocal Microscope (Specified in chapter 2.3), observing their surface roughness. For each process, 2 specimens were examined in 500x and 1500x magnification specified on table 16, focusing on their Surface roughness Sa (in fig 34) and Multi-line roughness Ra (in fig 35). Measuring both surface roughness Sa and multi-line roughness Ra serves more accurate evaluation of obtained surface roughness values.

Table 18 - Objectives and measurement parameters for surface roughness measurements:

Objective	Magnification	NA	WD [mm]	Zoom	(Lighting) Coaxial	(Lighting) Ring
50X Plan Apo	50x	0,95	0,35	1.0X	100 %	OFF
150X Plan Apo	150x	0,95	0,2	1.0X	100 %	OFF

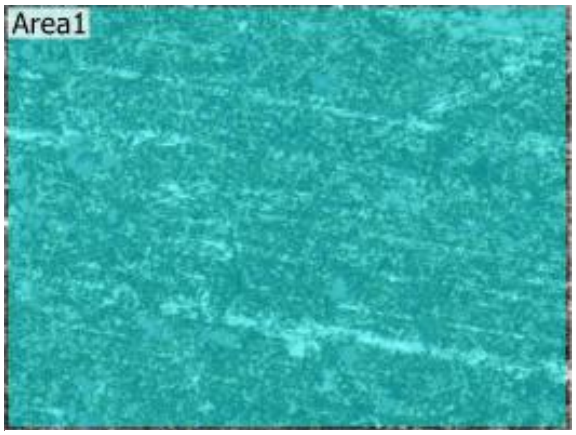


Fig. 34 - Surface roughness Sa

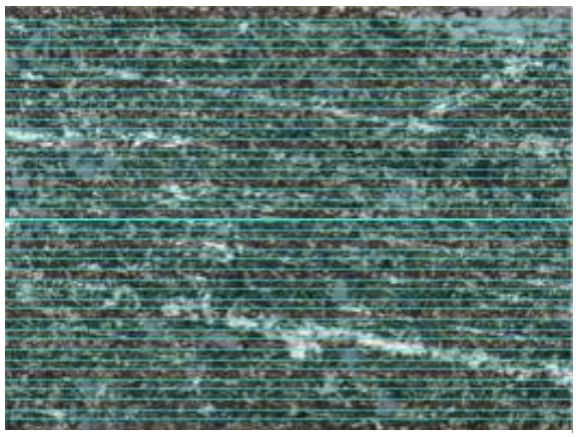


Fig. 35 - Multi-line roughness Ra

As result of this analysis, we were given surface roughness values in these 2 magnifications (500x,1500x) shown on table 17.

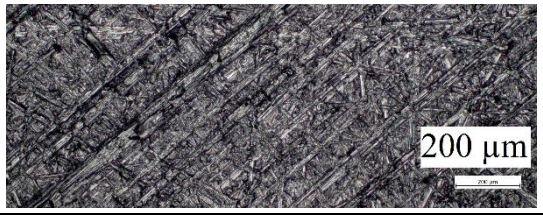
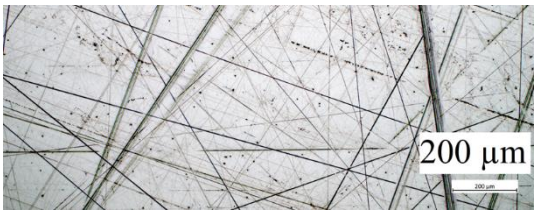
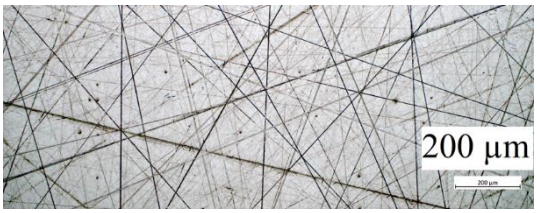
Table 19 - Surface and Multi-line roughness values

Surface Sa [um]						
Step \ Magnification	**50x**	**50x Average**	**150x (1)**	**150x (2)**	**150x (3)**	**150x Average**
SiC 01	0,117	**0,158**	0,063	0,070	0,075	**0,082**
SiC 02	0,198		0,112	0,107	0,067	
Largo 01	0,077	**0,074**	0,055	0,063	0,065	**0,062**
Largo 02	0,071		0,057	0,066	0,066	
Multi Line Ra [um]						
Step \ Magnification	**50x**	**50x Average**	**150x (1)**	**150x (2)**	**150x (3)**	**150x Average**
SiC 01	0,111	**0,153**	0,060	0,066	0,074	**0,078**
SiC 02	0,194		0,099	0,106	0,065	
Largo 01	0,070	**0,069**	0,054	0,062	0,064	**0,061**
Largo 02	0,067		0,056	0,065	0,065	

Surface roughness comparison shows us that using MD-Largo wheel gives us better surface roughness after these few grinding steps. Another thing to mention is that using one MD-Largo wheel is less time consuming than using 3 SiC wheels. Based on this information, we have chosen MTA aka Process 4: Struers Metalog Guide A as the most suitable polishing process.

With this process selected as the most suitable, we examined the prepared surface after every preparation step to see, how the surface changes. Examination was done on Zeiss Axio Observer 7 optical microscope (specified in chapter 2.3).

Table 20 - Step by step surface quality of MTA process

Step	Surface
SiC 220 grit	
MD-Largo DiaDuo 9 µm suspension	
MD-Mol 3 µm diamond paste	
MD-Dac 1 µm diamond paste	
MD-Dac 0,25 µm diamond paste	
MD-Chem 70 % OP-S + 30 % H2O suspension	

We also decided to try the exact same MTA process, but in the last step changed colloidal silica polishing time from 3 to 30 minutes to see, if this time increase gives us a better surface to examine. Results are shown in figures 36 and 37 with 1000x and 5000x magnification

Table 21 - 1000x, 5000x magnification measurement parameters

HV	Current	Detector	Mode	Magnification
1.00 kV	0,10 nA	ETD	SE	1000x, 5000x

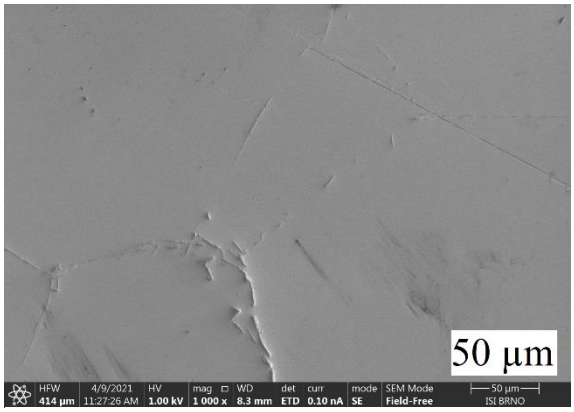


Fig. 36 - MTA 1000x, OP-S 30 min

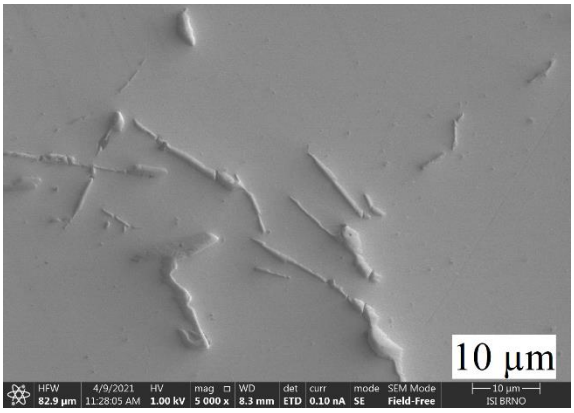


Fig. 37 - MTA 5000x, OP-S 30 min

Direct comparison showing the difference of using colloidal silicas for 3 minutes (fig 38) and for 30 minutes (fig 37). This comparison shows that polishing with OP-S for 3 minutes is not sufficient and OP-S polishing for 30 minutes gives us way better results.

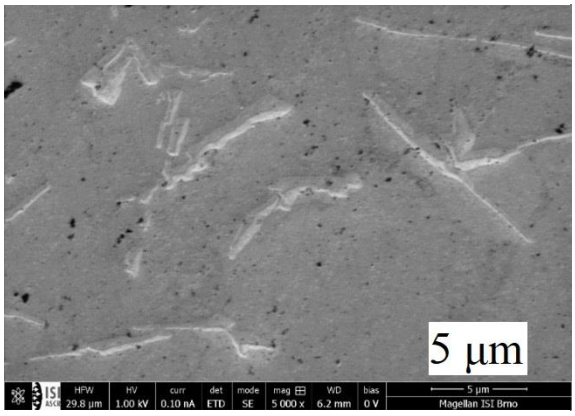


Fig. 38 - MTA 5000x, OP-S 3 min

To conclude colloidal silica polishing chapter, we found out that OP-U is not sufficient and OP-S is suitable when polishing pure aluminium for SEM observation. Another important fact is knowledge that colloidal silica polishing is producing better results with longer polishing times, namely 30 minutes in our case. Table 22 presents the most suitable and optimized process:

Optimal colloidal silica process:

Table 22 - Modified Process 4: Struers Metalog Guide A + colloidal silicas

Ste-p	Grind. paper	Wat-er	Lubricant	Suspension	Lo-ad [N]	Time [s]	Platen speed [rpm]	Head speed [rpm]	Rota-tion
1	SiC 220 Grit	Yes	-	-	20	60 s	300	150	Co-rot.
2	MD-Largo	-	-	DiaDuo 9 µm	10	180 s	150	100	Co-rot.
3	MD-Mol	-	IPA	DP Paste P 3 µm	5	240 s	200	100	Co-rot.
4	MD-Dac	-	IPA	DP Paste-P 1 µm	5	120 s	200	100	Co-rot.
5	MD-Dac	-	IPA	DP Paste-P 0,25 µm	5	120 s	200	100	Co-rot.
6	MD-Chem	-	-	70% OP-S 30 %H2O	5	30 min	150	100	Counter-rot.

Using colloidal silica polishing gave us very good results and important insight into the behavior of aluminium. This method on its own is fully proficient preparation method capable of preparing pure aluminium for SEM. Experience acquired in this chapter will be an ideal starting point for experimenting with electropolishing processes and finding out different approach for preparation of pure aluminium.

3.4 Electropolishing

Based on previous experiments, specimens were prepared by Process 4: Struers Metalog Guide A (MTA process), 4 of them done by 0,25 µm DP Paste-P as last step (in specimen name „M“) and other 11 done by colloidal silica suspension 70 % OP-S, 30 % H2O (in specimen name „S“ and „EL“) for 3 minutes as last step before using electropolishing.

We conducted electropolishing experiments on Struers Lectropol-5 (specified in chapter 2.2) with 15 specimens using different parameters shown on table 20. Based on recommended parameters in chapter 1.2.3 (page 10), we have chosen voltage values: **25 V, 39 V** and **50 V** and temperatures **15** and **22 °C.** The flow rate for all experiments was set to **10**. Recommended time of 120 s for perchloric-acetic acid electrolyte was cut down to **60** and **20 s**. This was done for 2 reasons, first of them is the full preparation by 0,25 µm (Dia paste or Silicas), which is finer than recommended preparation „3/0 paper“ (using SiC papers from coarse to fine). Other reason being experience from previous experiments with electropolishing aluminium. Electropolishing times for A2 electrolyte were spanned between **15-30 s.** Polished area for all specimens was **1 cm^2**

Table 23 - Electropolishing experiment parameters

Electrolyte	Specimen	Voltage [V]	Time [s]	Current [A]	Electrolyte temperature [°C]
Perchlor. + Acetic	12M	25	20	0,19	15
Perchlor. + Acetic	11M	25	60	0,13	15
Perchlor. + Acetic	05M	50	20	0,43	15
Perchlor. + Acetic	06M	50	60	0,34	15
Perchlor. + Acetic	01S	25	20	0,12	15
Perchlor. + Acetic	02S	25	60	0,19	15
Perchlor. + Acetic	03S	50	20	0,34	15
Perchlor. + Acetic	04S	50	60	0,32	15
A2	EL01	25	15	1,43	22
A2	EL02	39	15	1,94	22
A2	EL03	25	20	1,27	22
A2	EL04	39	20	2,12	22
A2	EL05	25	25	1,12	22
A2	EL06	25	30	1,82	22
A2	EL07	39	30	1,60	22

After the electropolishing processes, all of the specimens were observed in the Magellan 400 electron microscope. First of all, we compared differences in previous preparation before electropolishing. As mentioned before, processes marked „M“ were prepared just mechanically with 0,25 Dia paste and processes marked „S“ had final touch of colloidal silicas for 3 minutes. It was experimentally found that there were no major differences of surface quality after electropolishing depending on the previous preparation steps. Similarities are shown in figures 39 and 40 with magnification 5000x (1 kV, 0.1 nA, ETD detector, SE mode).

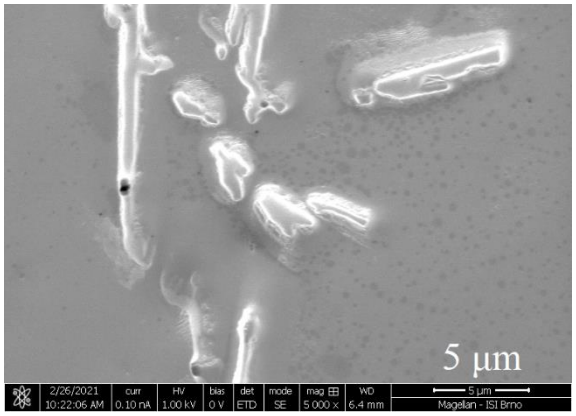


Fig. 39 – 12M 5000x – last step before electropolishing = 0,25 µm diamond paste

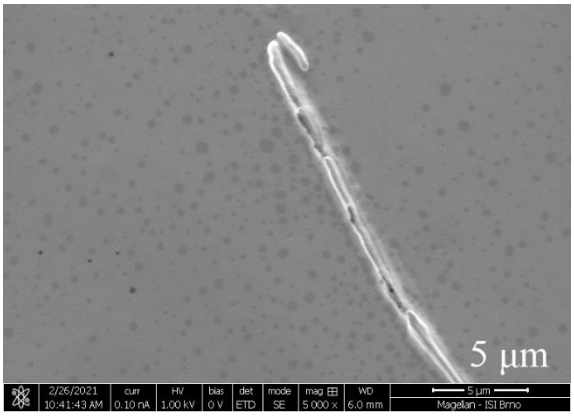


Fig. 40 – 01S 5000x – last step before electropolishing = OP-S 3 min

This means that mechanical preparation in combination with electropolishing is enough to prepare the surface without the use of colloidal silicas. We can therefore prepare specimen mechanically and then use short process of electropolishing. This saves a lot of time, especially when optimal colloidal silica process runs 30 minutes.

Most notable images of Perchloric-Acetic acid electrolyte are shown below, concretely specimens 01S, 02S and 03S with their respective parameters in 1000x and 5000x magnification.

Table 24 - 1000x magnification measurement parameters

HV	Current	Detector	Mode	Magnification
1.00 kV	0,10 nA	ETD	SE	1000x

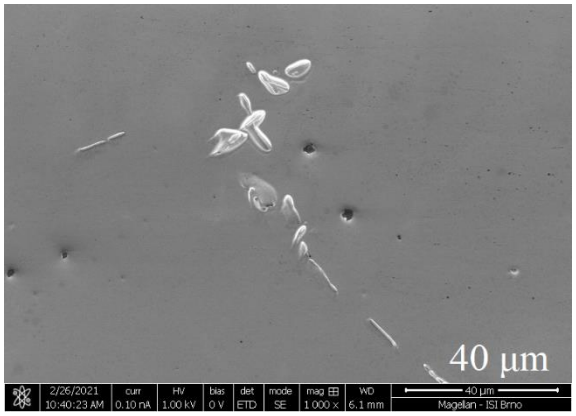


Fig. 41 – 01S 1000x, 25 V, 20 s

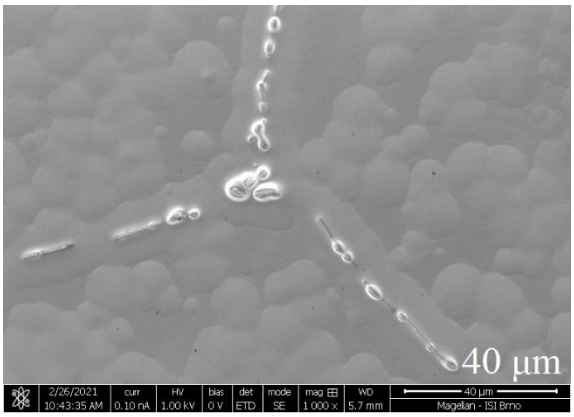


Fig. 42 – 02S 1000x, 25 V, 60 s

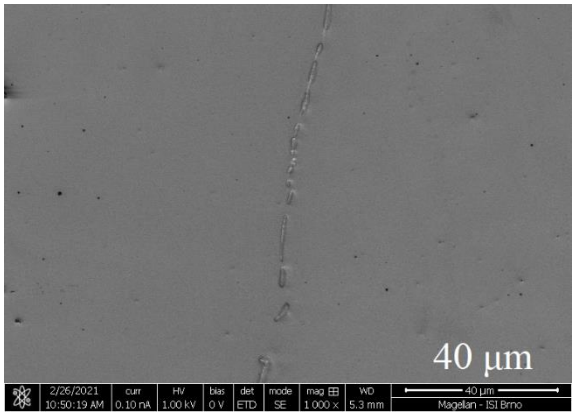


Fig. 43 – 03S 1000x, 50 V, 20 s

Table 25 - 5000x magnification measurement parameters

HV	Current	Detector	Mode	Magnification
1.00 kV	0,10 nA	ETD	SE	5000x

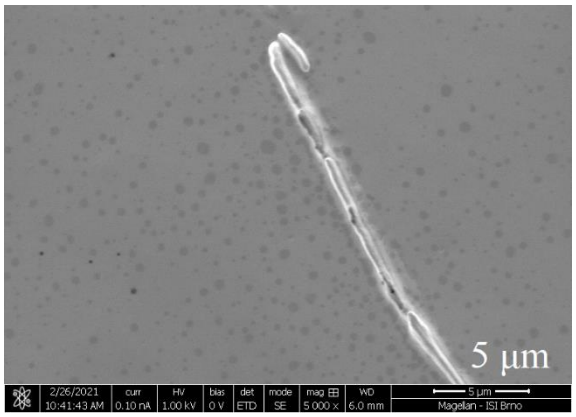


Fig. 44 – 01S 5000x, 25 V, 20 s

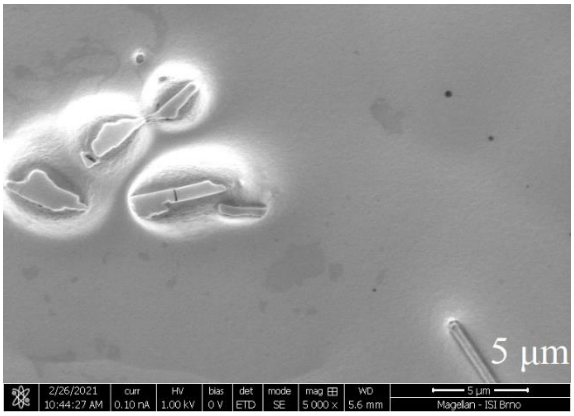


Fig. 45 – 02S 5000x, 25 V, 60 s

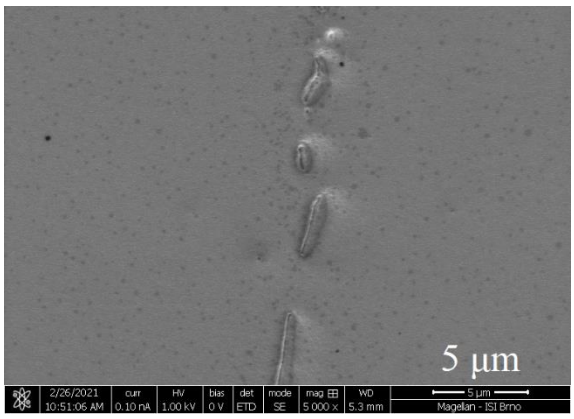


Fig. 46 – 03S 5000x, 50 V, 20 s

Images of specimens 01S, 02S and 03S were compared above in figures 41–46. Firstly, we will look at the 03S specimen, prepared with t = 20 s and U = 50 V. This specimen shows clean matrix and precipitates very well. These parameters are good for future use. When we look at 02S specimen in figure 42, we see that electropolishing process consisting of t = 60 s and U=25 V over-etched the surface, making craters into the matrix and over-etching the β-AlFeSi precipitates. This combination of parameters is therefore not suitable. Specimen 01S, prepared with parameters t = 20 s and U = 25 V, is also showing good results, having clean matrix and clearly visible β-AlFeSi precipitates. This is giving us glimpse into how electropolishing time affects the surface. Most optimal times so far are around 20 s.

With experience and knowledge from using perchloric-acetic acid electrolyte, we moved onto Struers A2 electrolyte. Best results were showed on specimens EL01, EL03 and EL05. These specimens show similarities in their parameters. All of these three being prepared at 25 V voltage and gradually increased time by 5 s. Concretely 15, 20 and 25 seconds. These specimens are shown in figures below.

Table 26 - 1000x magnification measurement parameters

HV	Current	Detector	Mode	Magnification
1.00 kV	0,10 nA	ETD	SE	1000x

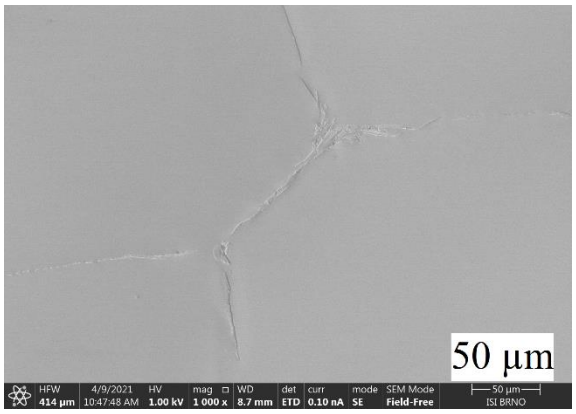


Fig. 47 – EL01 1000x, 25 V, 15 s

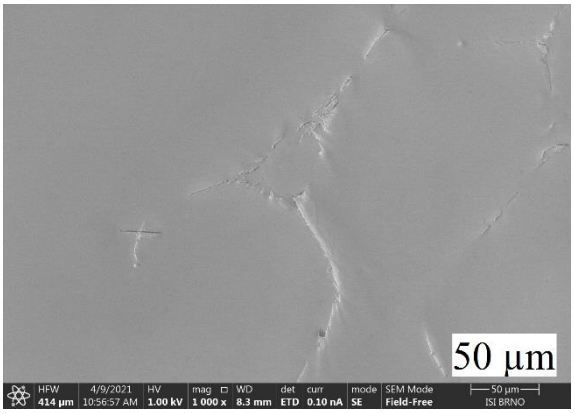


Fig. 48 – EL03 1000x, 25 V, 20 s

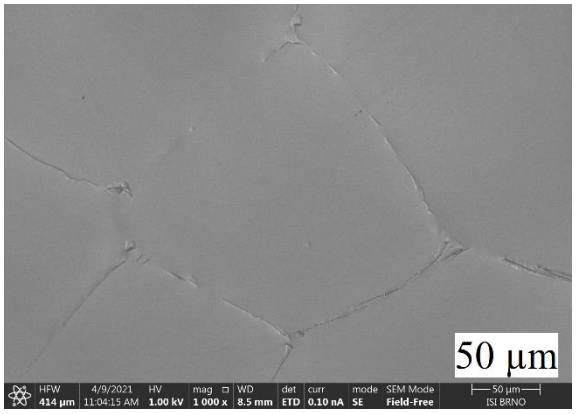


Fig. 49 – EL05 1000x, 25 V, 25 s

Table 27 - 5000x magnification measurement parameters

HV	Current	Detector	Mode	Magnification
1.00 kV	0,10 nA	ETD	SE	5000x

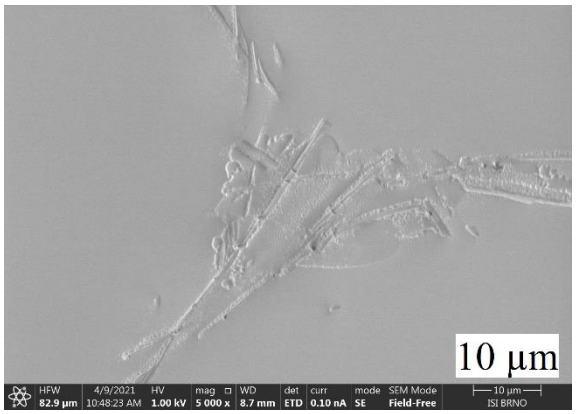


Fig. 50 – EL01 5000x, 25 V, 15 s

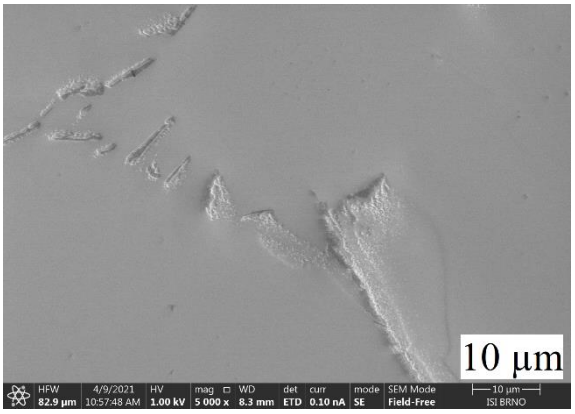


Fig. 51 – EL03 5000x, 25 V, 20 s

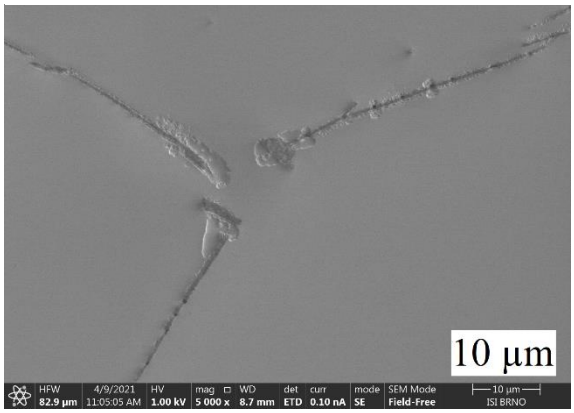


Fig. 52 – EL05 5000x, 25 V, 25 s

We can see that all three specimens prepared with **25 V** with times **15, 20 and 25 s** are very good. With altering times, we can alter how much the precipitates and grain boundaries will be etched. Specimen EL01 (15 s) is revealing precipitates a little bit. Specimen EL03 (20 s) is best suited for revealing precipitates and specimen EL05 (25 s) in magnification 1000x is enabling us to view grain boundaries very well. Other specimens, with higher voltages were overetched or had craters in matrix or precipitates. In figures 53 and 54 are shown specimens EL02 and EL04 at 5000x magnification, which have voltage **39 V** and times **15 s** and **20 s**, clearly being overetched at such short times.

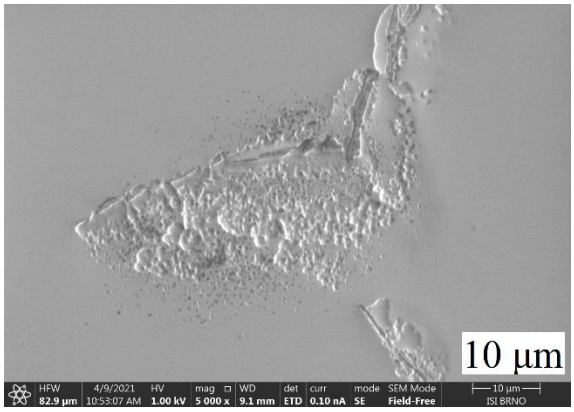


Fig. 53 – EL02 5000x, 39 V, 15 s

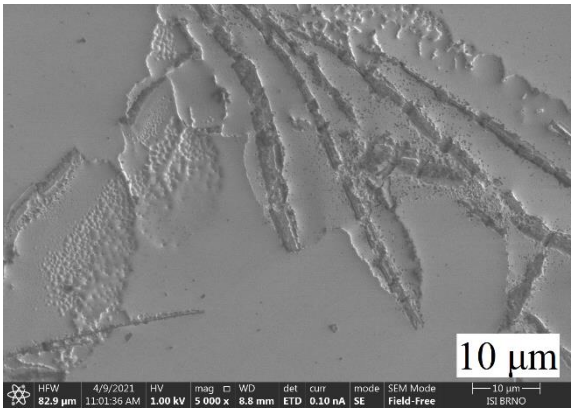


Fig. 54 – EL04 5000x, 39 V, 20 s

After experimenting with different electrolytes, voltages and times, we have found that before electropolishing, previous preparation can be just mechanical, which saves preparation time instead of using time consuming colloidal silicas. As optimal electrolyte, Struers A2 electrolyte was chosen. A2 electrolyte shows better results in SEM, is more commercially available, easier to prepare and not as dangerous as perchloric-acetic acid electrolyte. A2 electrolyte is also good for using at room temperatures. Optimal voltage value, which produced best results without degrading the surface and making craters, was 25 V. Optimal electropolishing times range from 15 to 25 s depending on how much etched surface we want to produce.

Optimal electropolishing process is shown on table 28.

Optimal electropolishing process:

Table 28 - Modified Process 4: Struers Metalog Guide A + electropolishing

Ste-p	Grind. paper	Wat-er	Lubricant	Suspension	Lo-ad [N]	Time [s]	Platen speed [rpm]	Head speed [rpm]	Rota-tion
1	SiC 220 Grit	Yes	-	-	20	60 s	300	150	Co-rot.
2	MD-Largo	-	-	DiaDuo 9 µm	10	180 s	150	100	Co-rot.
3	MD-Mol	-	IPA	DP Paste P 3 µm	5	240 s	200	100	Co-rot.
4	MD-Dac	-	IPA	DP Paste-P 1 µm	5	120 s	200	100	Co-rot.
5	MD-Dac	-	IPA	DP Paste-P 0,25 µm	5	120 s	200	100	Co-rot.
6	Electropolishing = A2 electrolyte, 22 °C, 25 V, 15-25 s, Flow rate 10, Area 1 cm^2								

Electropolishing processes showed us different approach to prepare specimen for SEM microscopy, with main advantage being cutting the overall preparation time. These processes are also sufficient for SEM purposes.

4 Conclusion

First of all, precise sectioning (i.e. cutting) of specimen is needed to avoid introduction of deformations and prevent thermal impact to the area of the cut. After the specimen is sectioned and mounted into the resin, the mechanical grinding process can start. Based on performed experiments, the mechanical processes ending with the finest available diamond paste (0,25 μm abrasive grain size) are macroscopically scratched and do not exhibit sufficient quality for purposes of microscopy characterization. The solely mechanical polishing is therefore not sufficient for scanning electron microscopy. The mechanical polishing is a cornerstone for advanced preparation methods, such as polishing using the colloidal silicas or the electrolytic polishing. Experimental results showed that using of the OP-U suspension results in contamination and degrading of the surface. The OP-S suspension seems to be more optimal for our cause i.e. preparing pure aluminium. Regarding the polishing time, 3 minutes are not sufficient and longer time (about 30 minutes) leads to better results and the specimens suitable for SEM are obtained. An alternative approach to colloidal silica polishing is an electropolishing. Experiments showed that there is no need to prepare specimen with the colloidal silicas before the electropolishing. By other words, combination of the mechanical polishing and the electropolishing is sufficient. As the best electrolyte, the commercially available A2 electrolyte produced by Struers was chosen as optimal. Voltage of 25 V and 15-25 seconds of polishing time proved as the most optimal parameters for the electropolishing of pure aluminium. Specimens suitable for electron microscopy were obtained using above mentioned parameters.

Two most optimal preparation processes for producing pure aluminium metallographic samples suitable for the SEM observation are showed in tables 22 and 23. The first process combines the mechanical preparation with colloidal silicas polishing for 30 minutes. This process is more time consuming, but might come in handy, when a laboratory lacks the electropolishing equipment. The second process, which combines the mechanical preparation with the electropolishing is faster but requires the electropolishing equipment.

Optimal colloidal silica process:

Table 29 - Modified Process 4: Struers Metalog Guide A + colloidal silicas

Ste-p	Grind. paper	Wat-er	Lubricant	Suspension	Lo-ad [N]	Time [s]	Platen speed [rpm]	Head speed [rpm]	Rota-tion
1	SiC 220 Grit	Yes	-	-	20	60 s	300	150	Co-rot.
2	MD-Largo	-	-	DiaDuo 9 µm	10	180 s	150	100	Co-rot.
3	MD-Mol	-	IPA	DP Paste P 3 µm	5	240 s	200	100	Co-rot.
4	MD-Dac	-	IPA	DP Paste-P 1 µm	5	120 s	200	100	Co-rot.
5	MD-Dac	-	IPA	DP Paste-P 0,25 µm	5	120 s	200	100	Co-rot.
6	MD-Chem	-	-	70% OP-S 30% H2O	5	30 min	150	100	Counter-rot.

Optimal electropolishing process:

Table 30 - Modified Process 4: Struers Metalog Guide A + electropolishing

Ste-p	Grind. paper	Wat-er	Lubricant	Suspension	Lo-ad [N]	Time [s]	Platen speed [rpm]	Head speed [rpm]	Rota-tion
1	SiC 220 Grit	Yes	-	-	20	60 s	300	150	Co-rot.
2	MD-Largo	-	-	DiaDuo 9 µm	10	180 s	150	100	Co-rot.
3	MD-Mol	-	IPA	DP Paste P 3 µm	5	240 s	200	100	Co-rot.
4	MD-Dac	-	IPA	DP Paste-P 1 µm	5	120 s	200	100	Co-rot.
5	MD-Dac	-	IPA	DP Paste-P 0,25 µm	5	120 s	200	100	Co-rot.
6	Electropolishing = A2 electrolyte, 22 °C, 25 V, 15-25 s, Flow rate 10, Area 1 cm^2								

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6 List of used symbols, abbreviations

2D – 2 Dimensional

3D – 3 Dimensional

Al - Aluminium

$Al_{15}(Fe,Mn)_3Si_2$ - α-AlFeSi precipitate

Al_2O_3 - Aluminium oxide

Al_5FeSi - β-AlFeSi precipitate

ALT - Modified Process 3: Struers Aluminium Method 2 (Alternative)

Ba - Barium

BSE – Back Scattered Electron

C – Mass fraction of the constituent

CL - Cathodoluminescence

Cu - Copper

E(0) [eV] – Initial energy of electron in SEM

EBSD - Electron Backscatter Diffraction

EDS – Electron Dispersive X-Ray Spectroscopy

ETD - Everhart-Thornley Detector

FCC – Face Centered Cubic crystal lattice

Fe - Iron

FR [-] – Flow Rate

H_2O - Water

HAZ – Heat Affected Zones

HV – Accelerating voltage for the electron in SEM

IGA – Intergranular Attack

IPA - Isopropanol

Li - Lithium

M(final) [-] – Final Magnification

M(obj) [-] – Magnification of the objective

M(oc) [-] - Magnification of the ocular

MAIN - Modified Process 2: Struers Aluminium Method 1

MD – Magnetic Disc

Mg - Magnesium

MgO - Magnesium Oxide

Mn - Manganum

Mo - Molybdenum

MTA - Modified Process 4: Struers Metalog Guide A

N/D – Not Defined

NA - Numerical Aperture

Pb - Lead

pH – Potential of Hydrogen

Ra – Multi-line surface roughness

S - Sulfur

Sa – Surface roughness

SE – Secondary Electron

SEM – Scanning Electron Microscopy

Si - Silicon

SiC – Silicon Carbide

SiO_2 - Silicon Dioxide

STEM - Scanning Transmission Electron Microscopy

Ti - Titanium

VDW - Modified Process 1: Van der Woort process

WD – Working Distance

Z – Atomic number

Zn - Zinc

7 List of figures

8 List of tables